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VACUUM STANDARDS

IN OUR ISSUE Vol. II No. 3 (published in September 1953) we reported the formation, by the British Standards Institution, of a committee for the standardisation of Vacuum Engineering Terminology and went on to discuss the desirability of extending this in due course to other standardisation measures, particularly those covering performance claims and the test procedures involved. As was to be expected, the complexity of the subject, long-established customs and the difficulty of co-ordinating the work and ideas of interested vacuum workers in various sciences in different countries have had their effect on the speed with which the work on the various desirable standardisation aspects has progressed. The first task the British Standards Institution has undertaken, namely the standardisation of Vacuum Terminology has reached the stage where drafts have been circulated to interested people. The American Committee on Vacuum Techniques (CVT) has reached about the same position, as far as terminology is concerned. Meanwhile, the British Standards Institution has proceeded, primarily at the request of the chemical industry, to set up another technical committee to consider the Testing of Chemical Vacuum Plant, thus providing a valuable code, between manufacturers and customers, for the building of such equipment. At the same time, this code will undoubtedly serve to bring to the attention of chemical engineers the advanced developments that have taken place in vacuum techniques for leak detection.

Not forgetting the Société Française des Ingénieurs Techniciens du Vide, who are working in a similar direction, it is to the credit of the American CVT that they have carried equipment standardisation one step further by tackling the problem of pipe flanges. Proposals have been worked out, details of which are made known in this issue, for standardisation similar to that established already for other engineering applications of flanges and we very much hope that the call for comments which has been made by the Chairman of this particular committee of the CVT will meet with encouraging response.

However, while there are many obvious advantages in having equipment standardisation leading to interchangeability, we must be on our guard that lack of early encouragement for this large undertaking does not deter us from pursuing the standardisation considerations of really fundamental matters; we refer, in particular, to the standardisation of measuring techniques involved in the performance rating of vacuum equipment. Here is an outstanding example of the absence of internationally agreed testing procedures resulting in claims which, although they may be completely valid, do not serve the user for true evaluation. Methods of testing and performance claims based upon methods which could be excused in the early development days of vacuum techniques have become indefensible in view of the wealth of information now available on this subject and our present-day knowledge of the shortcomings and limitations of some of the earlier procedures. This matter becomes all the more important in the light of the growing need for vacuum equipment in large-scale industrial operations together with the entry of many new manufacturers of vacuum equipment.

Fortunately it is becoming increasingly appreciated by the leading vacuum equipment manufacturers that it is in their own interests and to that of the general advancement of vacuum engineering to see that such standards are brought into being and informal discussions are already in progress which we hope will soon lead to practical implementation.

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Vol. 4 195

Dr. Weinstein was born in London in 1916. He received his general education at London County Council elementary and secondary technical schools and his initial professional education at evening institutes and by private study. During this time he gained varied practical experience working as a laboratory assistant, glass blower and instrument maker.

In 1942 he took his B.Sc. in mathematics at the University of London. Subsequently he became a member of the staff of Messrs. Hilger and Watts, scientific equipment manufacturers, where he stayed till 1948 and was concerned with the design and development of a number of optical instruments. Dr. Weinstein completed his thesis on optical aberration theory which he wrote under the guidance of Professor L. C. Martin, in 1950 and was awarded the Ph.D. (London).

In 1951 Dr. Weinstein was appointed to his present post. In 1954, as the recipient of a Fulbright grant, he spent six months as a guest worker in the United States in the Optics and Metrology Division of the National Bureau of Standards.

Dr. Weinstein has lately become very well known as an expert on the theory of thin film optics. He has published more than twenty papers on this subject and various other aspects of applied optics such as lens aberration theory.

Dr. Weinstein is a Fellow of the Physical Society and serves as the Honorary Secretary of the Optical Group of that Society. Also, he is a member of the London Mathematical Society and, being actively interested in matters of education generally, has been appointed a member of the managing body of a group of London County Council primary schools.

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COMPUTATIONS IN THIN FILM OPTICS

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Summary

A SURVEY IS GIVEN OF computing methods in thin film optics and the merits of different schemes for different purposes are discussed. Some design techniques for multi-layer filters are described.

Sommaire

UN EXAMEN DES méthodes de calcul sur les couches minces optiques est donné, et les mérites de différents projets emplois sont discutés. Quelques projets techniques pour les filtres à multi-couches sont décrits.

ol.
4
954

INTRODUCTION

A MULTILAYER FILM, or for short, a multilayer is a pile of thin layers of transparent or light-absorbing materials, of large lateral dimensions compared with the thicknesses. 'Thin' signifies of the order of a wavelength of light.

Thin film optics as discussed here comprises the study of the way in which variations in wavelength, angle of incidence and state of polarisation of the incident light affect the transmission and reflection factors of multilayers. We will also consider the phase changes of the reflected and transmitted light, since these are important for some applications.

Thin film optics has assumed considerable importance since about 1945, when multilayer films began to be used for many purposes, *e.g.* narrow-band interference filters, broad-band filters, dichroic beam splitters and all-dielectric reflectors for Fabry-Perot interferometers. Several methods have been proposed for computing the properties of given multilayers and a few suggestions have been made for the design of multilayers of specified properties, *e.g.* a multilayer with a given variation of transmission with wavelength. The latter problem is more difficult, being an inverse problem, than the former in the same way and for the same reasons that integration is more difficult than differentiation and synthetic optical design is more difficult than ray-tracing. In this article a survey will be given of the different computing methods which have been proposed and an attempt will be made to suggest which are most appropriate to different problems and to different computing facilities. Design methods will also be discussed, although here the approach must be more tentative as the subject is still developing rapidly. No attempt will be made to give exhaustive references since bibliographies are given by Cotton and Rouard (1950), Mayer (1950) and Heavens (1955), but occasional references will be given when the source seems to be little known.

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DEFINITIONS AND NOTATION

Calculations in thin film optics are carried out under the assumption that the incident light is a plane polarised monochromatic plane wave of large lateral extent. The first assumption can be justified by showing that any desired type of radiation can be synthesised

by a combination of plane polarised monochromatic plane waves with suitable relations between them. The second assumption means that the multilayer is to be so large in diameter that diffraction effects at the edge of the aperture and the cutting down of multiple reflections by the edge of the multilayer can be neglected. In most cases this will be so, provided regions less than 0.1 mm. from the edge of the aperture are ignored.

The fundamental quantities in terms of which the calculations are carried out are the electric and magnetic field strengths in plane polarised monochromatic plane waves. For such a wave the electric field strength may be written

$$\mathcal{E} = \mathbf{E} \exp \frac{2\pi i}{\lambda} (ct - nx) \qquad (1)$$

Here x is the distance in the direction of propagation of the wave, n the refractive index of the medium, λ the vacuum wavelength, c the velocity of light and t the time. $\mathbf{E}$ is the electric field strength vector at zero time and distance. Strictly, it should be a vector with real components but we assume that a factor $\exp i\varphi$ which implies a phase retardation φ radians may be included in $\mathbf{E}$. Similarly the magnetic field strength is written

$$\mathcal{H} = \mathbf{H} \exp \frac{2\pi i}{\lambda} (ct - nx) \qquad (2)$$

The exponential representation of $\mathcal{E}$ and $\mathcal{H}$ implies that the real (or, if desired, the imaginary) part is taken. However, the exponential form satisfies the differential equations for $\mathcal{E}$ and $\mathcal{H}$ and on account of the linearity of these equations all operations can be conducted on the complex exponential form and the real (or imaginary) part taken at the end. This results in a considerable mathematical simplification.

For an isotropic medium it is known that $\mathcal{E}$ and $\mathcal{H}$ as given by (1) and (2) satisfy the relation

$$\mathcal{H} = n\,\mathbf{p} \times \mathcal{E} \qquad (3)$$

where $\mathbf{p}$ is the unit vector in the direction of propagation.

The illumination on a screen perpendicular to the wave defined by Equ. (1) is proportional to the power per unit area, *i.e.* the Poynting vector $\mathbf{S} = \mathbf{E} \times \mathbf{H}$, so that from Equ. (3) the illumination is

$$n \left| \mathbf{E} \right|^2 \qquad (4)$$

This is the quantity to be used in determining the reflection and transmission factors. On the other hand the intermediate computations will involve $\mathbf{E}$ and $\mathbf{H}$ and these will give the phase changes also.

For a given multilayer let the incident, reflected and transmitted electric vectors be $\mathbf{E}$, $\mathbf{E}''$ and $\mathbf{E}'$ respectively and the initial and final media be n and n'. Then the reflection factor is obviously

$$R = \left| \frac{\mathbf{E}''}{\mathbf{E}} \right|^2 \qquad (5)$$

The transmission factor would similarly appear to be

$$T_a = \frac{n'}{n} \left| \frac{\mathbf{E}'}{\mathbf{E}} \right|^2 \qquad (6)$$

This is the ratio of the illumination on test screens perpendicular to the rays on either side of the multilayer. We could also define the transmission factor as the ratio of the total light flux incident on, and transmitted by, a given area of the multilayer. If the initial and final angles of incidence are φ and φ' this is given by

$$T_b = \frac{n' \cos \varphi'}{n \cos \varphi} \left| \frac{\mathbf{E}'}{\mathbf{E}} \right|^2 \qquad (7)$$

Which definition is used depends on the purpose for which the multilayer is intended. Roughly, Equ. (6) would be used, if the multilayer is at a field stop in the optical system, Equ. (7) if it is at an aperture stop. We note that the definitions reduce to the same thing, if the light is incident normally or if $n = n'$, and also that T_b is the form to be used in applying energy considerations, *i.e.* $R + T_b = 1$ if there is no absorption in the multilayer.

In order to give detailed formulae for computing $\mathbf{E}'$ and $\mathbf{E}''$ it is necessary to have systematic notation for the electric and magnetic field strengths in each medium in the multilayer. Referring to Fig. 1, let there be $p+1$ media, of which media 0 and p bound the multilayer, and let the light be incident at an angle φ_p to the common normal from medium p.

Vol
4
195

Let the thickness of the j^{th} medium be h_j, let its refractive index be n_j and let the angle of incidence be φ_j. The formulae are conveniently put in terms of the resolved parts of the electric and magnetic field strength vectors parallel to the multilayer; these tangential components will be denoted by E and H with appropriate subscripts and superscripts. For incident light, polarised with the electric vector in the plane of incidence, the component E will lie in the plane of Fig. 1. In each of the media, except medium 0, will be two waves, one ' going ' and one ' returning '. We denote the tangential components of those in the j^{th} medium by E_j^+ and E_j^-. Similarly the corresponding magnetic components will be H_j^+ and H_j^-. In the figure the arrows refer to the ray directions, not to the directions of the components E_j^+ and E_j^-. Specifically $E_j^{\pm}$ $(j=1, 2 \ldots p)$ is defined as the instantaneous value of the tangential component of $\mathcal{E}_j^{\pm}$ at zero time and at the interface between the j^{th} and $j-1^{th}$ media. Similarly for $H_j^{\pm}$; E_o^+ is the instantaneous value at the 0–1 interface. E_j^+ is a complex number, of which the argument gives the phase, and the modulus gives the amplitude of the light disturbance.

No distinction is made between the E and H symbols for polarisations parallel and perpendicular to the plane of incidence because, as will be seen in the next section, the equations are formally the same if a certain simplifying convention is used.

FUNDAMENTAL RECURRENCE RELATIONS

The symbols defined in the previous section can be now used to express the basic relationships between the field strengths in neighbouring media of the multilayer. By repeated application of these relations the media inside the multilayer are eliminated and the final data are obtained for computing R and T.

The total tangential components of electric and magnetic field strength are continuous at an interface; this condition yields the following set of equations, from which the magnetic components have been eliminated :—

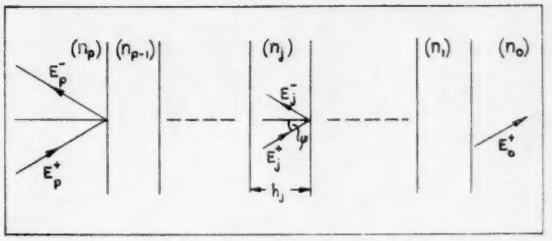


Fig. 1. Notation scheme

$$E_{j+1}^+ + E_{j+1}^- = E_j^+ e^{ig_j} + E_j^- e^{-ig_j}$$

$$u_{j+1}\{E_{j+1}^+ - E_{j+1}^-\} = u_j\{E_j^+ e^{ig_j} - E_j^- e^{-ig_j}\}$$

$$(j=0,1, \ldots p-1) \qquad (8)$$

where

$$g_j = \frac{2\pi}{\lambda} n_j h_j \cos \varphi_j \qquad (9)$$

and
or

$$\left.\begin{aligned} u_j &= n_j/\cos \varphi_j \\ u_j &= n_j \cos \varphi_j \end{aligned}\right\} \qquad (10)$$

dependent on whether the incident light is polarised with the electric vector in or perpendicular to the plane of incidence. In Equs. (8) it is to be understood that E_o^- and g_o are identically zero.

The quantities g_j are the effective optical thicknesses in radians of the respective media. The u_j may be taken as generalised refractive indices depending on the angle of incidence and the polarisation direction.

Equations (8) are a set of $2p$ homogeneous linear simultaneous equations in the $2p+1$ unknown $E_j^{\pm}$ and they are easily solved step-by-step in a number of ways, as will be shown in succeeding sections.

Equation (11)

	R	T_a	T_b
El. Vector in Plane of Incidence	$\left\|\frac{E_p^-}{E_p^+}\right\|^2$	$\frac{n_o \cos^2 \varphi_p \left\|E_o^+\right\|^2}{n_p \cos^2 \varphi_o \left\|E_p^+\right\|^2}$	$\frac{n_o \cos \varphi_p \left\|E_o^+\right\|^2}{n_p \cos \varphi_o \left\|E_p^+\right\|^2}$
El. Vector Perpendicular to Plane of Incidence	$\left\|\frac{E_p^-}{E_p^+}\right\|^2$	$\frac{n_o}{n_p}\left\|\frac{E_o^+}{E_p^+}\right\|^2$	$\frac{n_o \cos \varphi_p \left\|E_o^+\right\|^2}{n_p \cos \varphi_o \left\|E_p^+\right\|^2}$

Here we return to Equs. (5), (6) and (7). On account of the resolution into tangential components the expressions for R, T_a and T_b depend on the polarisation. The results are expressed by Equs (11) shown at the bottom of previous column.

At this stage it is convenient to note expressions for the phase changes on reflection and transmission, quantities which are useful in certain stages of designing and in some applications to interferometry. The phase change on reflection is arg (E_p^-/E_p^+) or, if E_p^-/E_p^+ is written as $x+iy$ where x and y are real, the phase change is arctan y/x. Similarly the phase change on transmission is arg (E_o^+/E_p^+). This, of course, refers to the phase difference between the transmitted light as it emerges from the last surface of the multilayer and the incident light as it strikes the first surface.

If, as is usually the case, the incident light is not plane polarised in or perpendicular to the plane of incidence, the reflection and transmission factors for the two polarisations can be suitably combined. In the simplest case, if the incident light is plane polarised at an angle θ to the plane of incidence it can be resolved into two fractions of which the amplitudes in the two directions are proportional to cos θ and sin θ. These fractions are incoherent in the sense that they cannot interfere, although their phases are coherent, so that the effects in the two directions are to be combined incoherently. The transmission factor is therefore $\cos^2\theta T_{\parallel}+\sin^2\theta T_{\perp}$, with an obvious notation. The emergent wave will in general be elliptically polarised and the axes of the ellipse can be found from the phase changes on transmission for the two components. If the incident light is elliptically polarised it must again be resolved into two plane polarised components with relative amplitudes say a and b; the initial phase difference is disregarded, since they are incoherent as far as the multilayer is concerned, and the final transmission coefficient is $(a^2T_{\parallel}+b^2T_{\perp})/(a^2+b^2)$. Finally natural or unpolarised light may be regarded as a random mixture of all possible polarisations. Thus the transmission factor is the mean over all azimuths of that for plane polarised light, *i.e.* it is $\frac{1}{2}(T_{\parallel}+T_{\perp})$; the transmitted light will in general be partially polarised.

SOLUTION OF THE RECURRENCE RELATIONS IN BASIC FORM

The solution of Equations (8) is given by (12) :—

$$E_{j+1}^{\pm}=\frac{1}{2u_{j+1}}\left\{(u_{j+1}\pm u_j)e^{ig_j}E_j^{+}+(u_{j+1}\mp u_j)e^{-ig_j}E_j^{-}\right\}$$

where the ambiguous signs have an obvious meaning and as before E_o^- and g_o are zero. Equations (12) are a set of recurrence relations, so that on assuming a given value for E_o^+, say unity, successive applications give $E_1^{\pm}$, $E_2^{\pm}$ .... $E_p^{\pm}$. Equations (11) can then be used to determine the reflection and transmission factors.

The Fresnel amplitude reflection and transmission coefficients at the $j+1^{th}$—j^{th} interface are $r_j=(u_{j+1}-u_j)/(u_{j+1}+u_j)$ and $t_j=2u_{j+1}/(u_{j+1}+u_j)$. Equ. (12) is sometimes written in terms of these :

$$\left.\begin{aligned}E_{j+1}^{+}&=\frac{1}{t_j}\left\{e^{ig_j}E_j^{+}+r_je^{-ig_j}E_j^{-}\right\}\\E_{j+1}^{-}&=\frac{1}{t_j}\left\{r_je^{ig_j}E_j^{+}+e^{-ig_j}E_j^{-}\right\}\end{aligned}\right\}\qquad(12a)$$

The basic solution provided by Equations (12) is not always the best suited for numerical computation. In the following sections a number of transformations using different variables will be considered and their advantages for different purposes will be discussed.

It is possible to obtain a solution of Equation (12) in closed form (Weinstein 1947) by carrying out the substitutions algebraically instead of numerically so that $E_p^{\pm}$ are given explicitly as functions of the u_j and g_j; the result is of little use for numerical work when there are more than four films in the multilayer since the number of terms is of order 2^{2p}, but it can be used for deriving general theorems.

Absorbing Films

In the derivation of Equations (12) it was tacitly assumed that none of the films was absorbing. The effect of absorption is allowed for by introducing a complex refractive index $\mathfrak{n}\equiv n-ik$. If this is substituted for n in Equ. (1) a real attenuation factor

Vol
4
195

$\exp(-2\pi kx/\lambda)$ appears, indicating that the complex amplitude is reduced by the factor $\exp(-2\pi k)$ per vacuum wavelength traversed by the wave in the direction of propagation. It can be shown that all of the equations so far derived are valid with the complex refractive index. The values of $\cos\varphi$ used in Equations (9) and (10) are obtained from Snell's law of refraction and they turn out to be complex. Ditchburn (1952) discusses the physical significance of complex angles of incidence. For the present purpose it is sufficient to note that the complex refractive indices for absorbing films can be substituted automatically in the computing formulae.

Matrix Expression of Equations (12)

The recurrence relations (12) and other relations which will be given subsequently can be written compactly in matrix notation. This is not merely an elegant variation but can result in a considerable saving of time in numerical computation, as will be seen. An explanation of the matrix notation and of the rules of matrix algebra as far as they are needed for this purpose is given in the Appendix. The matrix form of equation (12) is (13):—

$$\begin{pmatrix} E_{j+1}^{+} \\ E_{j+1}^{-} \end{pmatrix} = \frac{1}{2u_{j+1}} \begin{pmatrix} (u_{j-1}+u_j)e^{ig_j} & (u_{j+1}-u_j)e^{-ig_j} \\ (u_{j-1}-u_j)e^{ig_j} & (u_{j+1}+u_j)e^{-ig_j} \end{pmatrix} \begin{pmatrix} E_j^{+} \\ E_j^{-} \end{pmatrix}$$

or, if M_j denotes the square matrix on the right-hand side,

$$\begin{pmatrix} E_{j+1}^{+} \\ E_{j+1}^{-} \end{pmatrix} = \frac{1}{2u_{j+1}} M_j \begin{pmatrix} E_j^{+} \\ E_j^{-} \end{pmatrix} \tag{13a}$$

Substituting for the $E_j^{\pm}$ vector,

$$\begin{pmatrix} E_{j+1}^{+} \\ E_{j+1}^{-} \end{pmatrix} = \frac{1}{2u_{j+1}u_j} M_j M_{j-1} \begin{pmatrix} E_{j-1}^{+} \\ E_{j-1}^{-} \end{pmatrix} \tag{14}$$

and so finally

$$\begin{pmatrix} E_p^{+} \\ E_p^{-} \end{pmatrix} = \frac{M_{p-1}M_{p-2}\ldots M_2 M_1}{2^p u_p u_{p-1}\ldots u_2 u_1} \begin{pmatrix} (u_1+u_0)E_o^{+} \\ (u_1-u_0)E_o^{-} \end{pmatrix} \tag{15}$$

In this form each film is represented by a matrix, M_j, which does not depend on the values of $E_{j-1}^{\mp}$ and this has three advantages. First, the effect of a change in thickness or refractive index of the jth film can be calculated without re-calculating the whole multilayer; the partial products $M_p \ldots M_{j+1}$ and $M_{j-1} \ldots M_1$ are known from the initial computation and the new value of M_j can be inserted in the complete product, $(M_p \ldots M_{j+1})M_j(M_{j-1} \ldots M_1)$. Secondly, it often happens in the course of a design that the same arrangement of films, a sub-multilayer as it may be called, occurs more than once in the same design or in subsequent modifications of the design; in this case the matrix product for the sub-multilayer is simply transferred to the new position without re-computing. Finally, periodic sub-multilayers often occur, in which an arrangement of two or more films is repeated several times. In this case the matrix for n periods is the nth power of the matrix for the basic period and there are very simple methods of taking a power of a matrix. The matrix expression of the recurrence relations in the following section is particularly advantageous in these ways.

Solution in Terms of Total $\mathscr{E}$ and $\mathscr{H}$ Components

As mentioned above the recurrence relations in terms of $E_j^{\pm}$ are not always the most convenient for numerical work. An alternative equation is obtained by expressing (12) in terms of the variables E_j and H_j given by

$$\left.\begin{aligned} E_j &= E_j^{+} + E_j^{-} \\ H_j &= H_j^{+} + H_j^{-} \\ &= u_j\ (E_j^{+} - E_j^{-}) \end{aligned}\right\} \tag{16}$$

These are the total tangential components of the electric and magnetic field strength vectors; solving for $E_j^{\pm}$ yields

$$E_j^{\mp} = \tfrac{1}{2}\,(E_j \pm H_j/u_j) \tag{17}$$

When these are substituted in equations (12) the result is

$$\left.\begin{aligned} E_{j+1} &= E_j \cos g_j + \frac{i}{u_j} H_j \sin g_j \\ H_{j+1} &= i u_j E_j \sin g_j + H_j \cos g_j \end{aligned}\right\} \qquad (18)$$

or in matrix form

$$\begin{pmatrix} E_{j+1} \\ H_{j+1} \end{pmatrix} = \begin{pmatrix} \cos g_j & \frac{i}{u_j} \sin g_j \\ i u_j \sin g_j & \cos g_j \end{pmatrix} \begin{pmatrix} E_j \\ H_j \end{pmatrix} \qquad (19)$$

when the index j runs from 1 to $p-1$.

The initial values are given by $E_1 = E_o^+$ and $H_1 = u_o E_o^+$ and the values of $E_p^{\pm}$ are obtained from Equ. (17).

The recurrence relations (19) are usually the best for numerical computation with a desk calculating machine. When none of the films absorbs—which is a particularly frequent case—all of the u's and g's are real so that the leading diagonal elements of the matrix are real and the other elements are purely imaginary. It can easily be seen that the product of any number of such matrices also has real elements in the leading diagonal and pure imaginary elements elsewhere, so that the numerical work in forming a product of such matrices is no greater than that for a product of wholly real matrices. If a film has a complex refractive index, this advantage disappears and then it may be simpler to use Equs. (13) in order to avoid taking trigonometrical functions of complex quantities. A further advantage of Equations (19) lies in a very convenient numerical check : For any film, absorbing or not, the determinant of the matrix is equal to unity so that, since the determinant of a product of matrices is the product of their determinants, the determinant of the matrix of any multilayer is also unity. This provides a numerical check which can be applied at any stage in the formation of a continued product of matrices. A similar check is possible for the matrix of Equ. (13), but the determinant here is $2u_j$, if the factor $1/2u_{j+1}$ is included in the matrix.

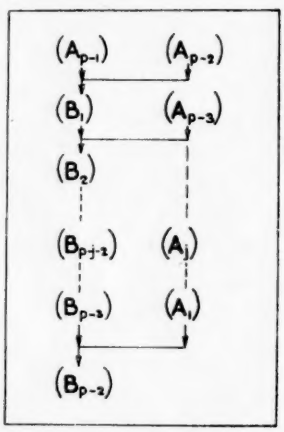


Fig. 2. Scheme for matrix products

The numerical work rarely needs to be done with more than four significant figures but an electrical desk calculator is useful since it permits writing down the product of two matrices without intermediate entries. Although the computation of a continued matrix product according to Equations (19) is quite straight-forward, it may be useful to describe the computing scheme adopted by the author as a result of a number of trials. The matrices $A_{p-1} \ldots A_1$ to be multiplied are written down in a column, except for A_{p-1}, as in Fig. 2. The product $A_{p-1}\, A_{p-2}$, say B_1, is written under A_{p-1}, $B_1\, A_{p-3}$ is written under B_1 and so on. This scheme has the advantages that the two matrices to be multiplied are side by side and that all the partial products starting from one end of the product are present. Thus if a change is made in A_j the partial product $A_{p-1} \ldots A_{j+1} \equiv B_{p-j-2}$ is already available. The other partial product $A_{j-1} \ldots A_1$ could be computed separately as a product but a better method is to divide the complete product B_{p-2} by B_{p-j-1} or pre-multiply it by the reciprocal of B_{p-j-1}.

The reciprocal of a matrix given by

$$a_{ij} \equiv \begin{pmatrix} a_{11} & a_{12} \\ a_{21} & a_{22} \end{pmatrix}$$

and having unit determinant, is

$$(a_{ij})^{-1} = \begin{pmatrix} a_{22} & -a_{12} \\ -a_{21} & a_{11} \end{pmatrix} \qquad (20)$$

so that the computation of the partial products is very simple.

The scheme in Fig. 2 must be carried out for each wavelength at which R and T are required, the values of the g_j's being suitably chosen. It is convenient to

Vol. 4 1954

start at some central wavelength λ_o, for which it often happens that the g's are multiples of $\pi/2$, and to obtain the g's for other wavelengths by applying a factor k to the g's; this factor is a kind of wave-number relative to λ_0. If the light is incident obliquely, a preliminary computation to determine the values of the various $\cos\theta$ and u is necessary, and this is repeated for each different angle of incidence but not each wavelength.

Recurrence Formulae for Amplitude Reflection and Transmission Factors

Immediately to the left of the interface between the jth and j–1th media we may define the amplitude reflection factor as the complex number

$$\mathcal{R}_j = \frac{E_j^-}{E_j^+} \tag{21}$$

and similarly the amplitude transmission factor as

$$\mathcal{T}_j = \frac{E_o^+}{E_j^+} \tag{22}$$

The following recurrence relations for these two quantities can be derived from Equations (12a)

$$\mathcal{R}_{j+1} = \frac{t_j + e^{-2ig_j}\mathcal{R}_j}{1 + r_j e^{-2ig_j}\mathcal{R}_j} \tag{23}$$

$$\mathcal{T}_{j+1} = \frac{t_j e^{-ig_j}\,\mathcal{T}_j}{1 + r_j e^{-2ig_j}\mathcal{R}_j} \tag{24}$$

The initial values are $\mathcal{R}_1 = r_o$ and $\mathcal{T}_1 = t_o$. It will be noticed that Equ. (23) contains only the variable $\mathcal{R}_j$ so that it is a single recurrence relation instead of two as in previous sections. This gain in simplicity is offset by several disadvantages, first Equ. (23) alone gives no information about transmission unless the films are non-absorbing, and thus Equations (23) and (24) must still be used in the most general case. Secondly, using Equ. (23) alone there is no possibility of a check formula, since no redundant variables are carried. Also, even with non-absorbing films, $\mathcal{R}_j$ and $\mathcal{T}_j$ are in general complex quantities so that the numerical work for the single Equ. (23) is no lighter than for Equ. (19). Finally the properties of a single film are no longer contained in a matrix and separate from the rest of the multilayer, so that if, a film or sub-multilayer is changed all the subsequent parts of the multilayer must be re-computed. Thus Equations (23) and (24) offer no particular advantages.

The Admittance Method and the Smith Chart

Another recurrence relation is obtainable in terms of the variable $y_j \equiv H_j/(u_j E_j)$ from Equations (18):

$$y_{j+1} = \left(\frac{u_j}{u_{j+1}}\right)\frac{i\sin g_j + y_j \cos g_j}{\cos g_j + iy_j \sin g_j} \tag{25}$$

The quantity y is known as the admittance (sometimes as the reduced admittance) since it has the dimensions of a reciprocal impedance and the formulae involving E, H, y^{-1} are similar in form to the voltage, current and impedance relationships in four-terminal networks. Equ. (25) is subject to much the same criticism as Equ. (23) as a practical computing equation but it leads to a very elegant graphical method, the Smith Chart. From Equations (23) and (24) it follows that

$$y_{j+1} = \frac{1-\mathcal{R}_{j+1}}{1+\mathcal{R}_{j+1}} = \frac{u_j}{u_{j+1}}\left(\frac{1-\mathcal{R}_j e^{-2ig_j}}{1+\mathcal{R}_j e^{-2ig_j}}\right) \tag{26}$$

The Smith chart, shown in Fig. 3, enables Equ. (26) to be computed graphically. The plane of the chart is the plane of the complex variable $\mathcal{R}$, for which a polar coordinate mesh with origin at the centre and real axis horizontal is to be imagined. The coordinate mesh actually given in the chart is that of the function y given by the conformal transformation

$$y = \frac{1-\mathcal{R}}{1+\mathcal{R}} \tag{27}$$

The mesh lines are for constant real and imaginary parts of y.

The following steps are used to go from $\mathcal{R}_j$ to $\mathcal{R}_{j+1}$:

(1) Plot $\mathcal{R}_j$ in the $\mathcal{R}$ coordinate mesh, with protractor and ruler.

(2) Rotate the $\mathcal{R}_j$ vector through an angle $2g_j$ radians; this gives $\mathcal{R}_j e^{-2ig_j}$.
(3) Read off the value of the point obtained from (2) in the y coordinate mesh; this gives

$$(1-\mathcal{R}_j e^{-2ig_j})/(1+\mathcal{R}_j e^{-2ig_j}).$$

(4) Multiply this by u_j/u_{j+1} (by slide-rule) and re-plot in the y-mesh; this gives y_{j+1}.
(5) The last point gives $\mathcal{R}_{j+1}$ in the $\mathcal{R}$-mesh; this would not normally be read off, step (2) would follow instead. When $\mathcal{R}_p$ is reached the squared modulus gives R_p.

The Smith chart, which is in considerable use by electrical engineers for electrical filter calculations, can be very rapid and it is accurate enough for a number of multilayer computations, although not for all. It is unsuitable where rapid changes in transmission or very low transmissions occur due to having a large number of films in the multilayer. It is not so fast for absorbing layers and in this case it has the disadvantage that the transmission factor is not obtained. An alternative form of the chart with a polar y coordinate mesh, *i.e.* lines of constant amplitude and argument, is sometimes used and is perhaps slightly more convenient. The form shown in Fig. 3 is available commercially with a rotating cursor.

To summarise, the matrix method using total $\mathcal{E}$ and $\mathcal{H}$ components (see page 8) is best for general purposes, the matrix method described on page 7 is slightly more convenient for absorbing films and the Smith chart is suitable where there are not many films in the multilayer and extreme values of T or $\delta T/\delta\lambda$ are not of great interest.

MULTILAYERS WITH PERIODIC STRUCTURE

The properties of a multilayer with a periodic structure can be computed by the methods described above. In particular, if the matrix method (see page 8) is used, the matrix for the fundamental period can be computed and raised to the appropriate power numerically. This is particularly simple where there are 2, 4, 8, . . . periods, since the square of a matrix can, with the aid of a calculating machine, be written down in one step, *i.e.* without having even to write the matrix itself down twice. For ordinary numerical work this is undoubtedly the quickest procedure. However, a closed expression for the nth power of a matrix is known and this enables a number of useful general properties of periodic structures to be predicted; it was first applied by Abelès (1948).

Let the matrix of the fundamental period shown on page 8 be

$$M \equiv \begin{pmatrix} a_{11} & a_{12} \\ a_{21} & a_{22} \end{pmatrix} \tag{28}$$

Then

$$M^n = \frac{1}{\lambda_1-\lambda_2}\begin{pmatrix} D_{11} & D_{12} \\ D_{21} & D_{22} \end{pmatrix} \tag{29}$$

where

$$\begin{aligned} D_{11} &= (\lambda_1{}^n-\lambda_2{}^n)a_{11}-(\lambda_1{}^{n-1}-\lambda_2{}^{n-1}) \\ D_{12} &= (\lambda_1{}^n-\lambda_2{}^n)a_{12} \\ D_{21} &= (\lambda_1{}^n-\lambda_2{}^n)a_{21} \\ D_{22} &= (\lambda_1{}^n-\lambda_2{}^n)a_{22}-(\lambda_1{}^{n-1}-\lambda_2{}^{n-1}) \end{aligned}$$

and λ_1 and λ_2 are the roots of the equation

$$\lambda^2-(a_{11}+a_{22})\lambda+1=0 \tag{30}$$

The roots can be expressed in the form $e^{\pm i\theta}$, where $\cos\theta=\frac{1}{2}(a_{11}+a_{22})$ and thus $\lambda_1{}^n-\lambda_2{}^n$ can be written as $2i\sin n\theta$ or $2\sin\mathrm{h}\, n\theta$ according as $\frac{1}{2}(a_{11}+a_{22})$ is less than or greater than unity in absolute magnitude.* When this is done it is possible to deduce the following properties of non-absorbing multilayers : At a wavelength such that $\frac{1}{2}|a_{11}+a_{22}|>1$ the reflectivity increases steadily with the number of periods and tends to unity as n tends to infinity. Such a zone, if it exists, is called a stopping or high-reflectivity zone. For the commonest case of fundamental period composed of two $\lambda/4$ layers of zinc sulphide and magnesium fluoride the stopping zone covers the relative wave-number range $0.833<\mathrm{k}<1.167$ which could be made to correspond to wavelengths 0.456μ to 0.642μ, say. Outside the stopping zone the transmission oscillates rapidly with wavelength, the

* Equations (29) and (30) are not valid for any 2×2 matrix M; the general formulae have been simplified by making use of the fact that the determinant of M is unity.

Vol. 4 195

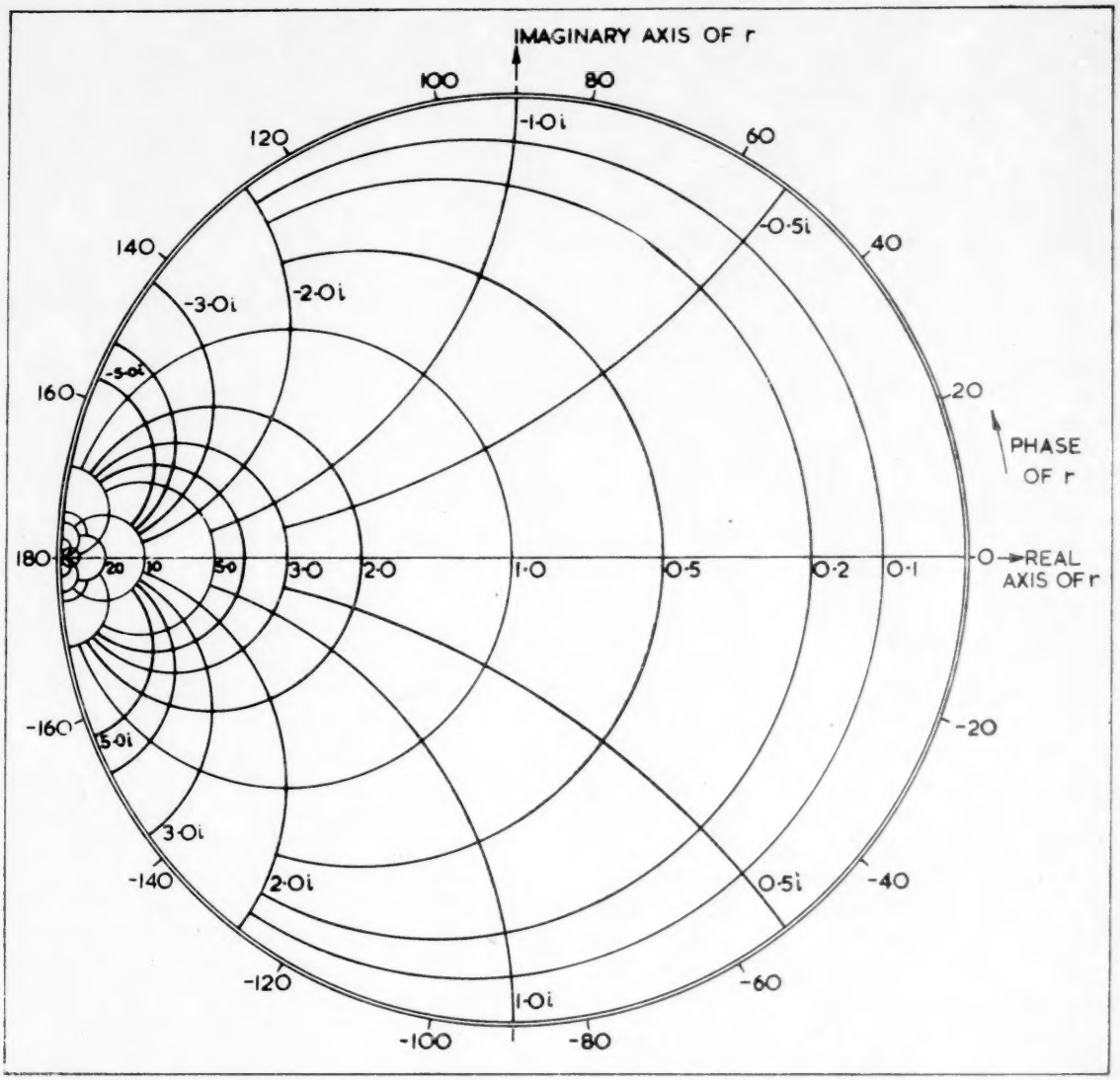


Fig. 3. The Smith Chart

frequency of the oscillations increasing with the number of layers. Fig. 4 shows the reflection factor for five $\lambda/4$ films of zinc sulphide separated by four of magnesium fluoride and bounded by the same medium on either side. Symmetrical structures of this kind have simpler properties than non-symmetrical ones. The limits of the high reflecting zone are shown by the dotted lines at $k=0.833$ and 1.167. Outside this zone the number of oscillations of R increases with the number of periods, there being one subsidiary maximum per period between $k=1.167$ and $k=2$; beyond $k=2$ the whole graph repeats

itself. As the number of periods varies, the subsidiary maxima touch an envelope which is shown dotted. Within the high-reflecting zone, R tends to unity as the number of periods increases. The phase change φ of the reflected beam is shown in the same figure. It has a finite derivative with respect to K at the centre of the high reflecting zone. The sign of this derivative is fortunately such that when these multilayers are used as reflectors in a narrow-band interference filter the order of the filter is effectively increased, so that a decreased half-width for given spacer-layer thickness is obtained. On the other hand the sensitivity of the filter to angle of incidence is not increased. The apparent jumps in φ occur at the zeros of R and the reason can be seen by reference to Fig. 5a, where the amplitude reflection factor $\mathcal{R}$ is plotted on the complex plane. Near a zero the extremity of $\mathcal{R}$ traverses the path shown in broken line, thus giving the jump in phase. These phase properties outside the high-reflecting zone are of importance in dichroic mirrors, where it is desirable to diminish the subsidiary maxima in order to obtain a more complete division of the spectrum. Some methods of doing this are indicated on page 14.

If the multilayer is made unsymmetrical through having different bounding media, the properties are slightly different from those shown in Fig. 4. The limits of the high-reflecting zone remain the same but the subsidiary maxima are decreased and the envelope is lower. Also, the minima are no longer zeros so that the other branch of the envelope for varying number of periods does not coincide with the k axis. The discontinuities in φ disappear, a portion of the new curve being typified by the long-chain line in the φ graph. In this case the amplitude reflection factor behaves as in Fig. 5b near a maximum of R and the transition from a to b justifies the plotting of φ as shown in Fig. 4, rather than as a steadily increasing function of k.

When the fundamental period consists of two films which are not of the same optical thickness changes of a similar kind occur. In addition, the width of the high-reflecting zone is always less than that for films of equal thickness and the subsidiary maxima are increased on one side of the high reflection zone and decreased on the other side; this gain has applications to dichroic mirrors. The same applies to the case of oblique incidence on a multilayer with equal thicknesses in the fundamental period. Fundamental periods with more than two films do not seem to have been investigated, but it seems quite possible that interesting properties such as wider high-reflecting zones might be obtainable.

MISCELLANEOUS COMPUTING METHODS

In this section some other approaches to the problem of thin-film computing which seem to the writer to be of limited applicability will be briefly described.

The Approximate Graphical Method

In this method the Fresnel reflection coefficient r_j (see page 6) for the $j+1$–j interface is plotted in the complex plane, with a rotation corresponding to twice the optical thickness of the films preceding it. The vector sum of these vectors for each interface gives an approximation for the amplitude reflection coefficient. The case of two films is illustrated in Fig. 6. The approximation is very crude, since no account is taken of multiple reflections in the multilayer or even of the decrease in amplitude of the light incident on the inner surfaces. The latter point can be covered by taking (see Fig. 6) $(1-|r_2|^2)^{\frac{1}{2}}r_1$, instead of r_1 and $(1-|r_2|^2)^{\frac{1}{2}}(1-|r_1|^2)^{\frac{1}{2}}r_0$ instead of r_0. The method is very useful where only a few films are involved, in particular for two- and three-film antireflection coatings, but it is quite unreliable for larger numbers of films and high reflection factors.

Explicit Formulae for R

It should be possible to extend the method in the previous paragraph and to obtain a series of terms, of which the first group would correspond to rays reflected once in the multilayer, the second to rays reflected three times, and so on. This would be an analogue of the usual method of obtaining the Airy summation for the Fabry-Pérot interferometer. Formulae which effectively do this were given by Schröder (1951) who started from the recurrence

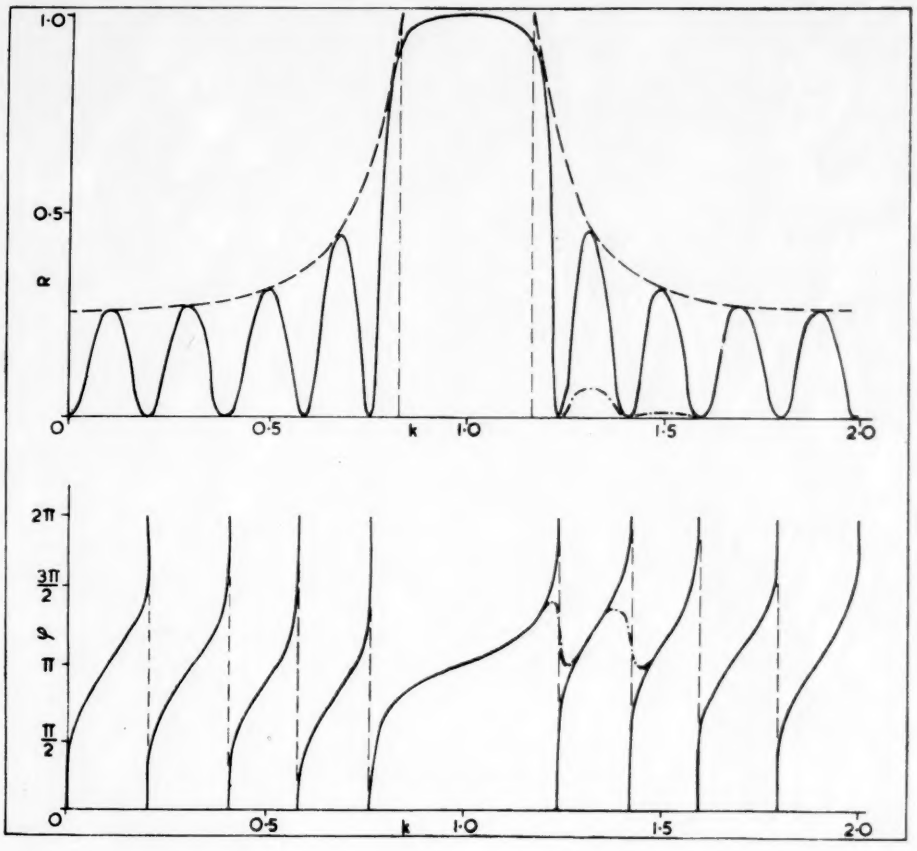
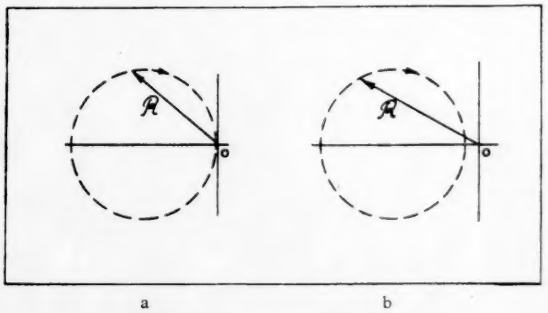


Fig. 4. Reflection factor and phase change for symmetrical multi-layer (5 high-index and 4 low-index-films).



a b

Fig. 5. Phase changes in reflected components.

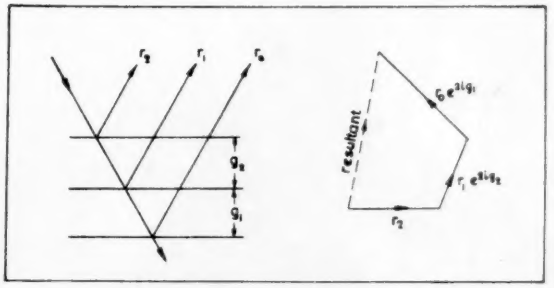


Fig. 6. Graphical approximation

Table 1. **Thin Film Constants**

Substance	*Wavelength* μ	*Refractive Index*	*Reflect. Factor*
MgF_2 (*aged*)†	.45 .5 .6 .7	1.374 1.369 1.367 1.366	
SiO_2	—	1.44	
$CaSiO_3$	.55	1.685	
Al_2O_3 (*anodic*)	.60	1.62	
Al_2O_3 (*heat oxidised Al*)	—	1.9	
SiO	.4 .5 .6 .7	$1.2-0.15i$ $2.0-0.02i$ 1.97 1.95	
Si	.405 .546 .589	$3.45-1.21i$ $3.95-0.57i$ $4.05-0.55i$	
CdS	.4 .5 .6 .7	$2.37-0.5i$ $2.53-0.15i$ $2.30-0.07i$ $2.24-0.05i$	
Ge	.405 .546 .578	$2.85-0.58i$ $3.47-0.40i$ $3.42-0.39i$	.35 .37 .36
ZnS	.45 .50 .60 .70	2.44 2.39 2.34 2.31	
TiO_2 (*Anodically oxidised Ti*)	.41 .51 .65 .8	2.55 2.42 2.30 2.26	
TiO_2 (*heat-oxidised Ti*)	.40 .50 .60 .70	3.00 2.79 2.67 2.61	
Ag*	.55	$0.1-0.39i$	0.95
Al	.4 .5 .6 .7	$0.38-3.9i$ $0.62-4.85i$ $0.97-5.851$ $1.35-7.0i$	0.9 0.9 0.9 0.9
Rh	—	$1.62-4.63i$	0.7

* The data for silver are very approximate since very large variations occur according to method of deposition and age of film.

† Only magnesium fluoride is listed as a low-index material because the other materials which have been used have refractive indices very little different from that of MgF_2 and their mechanical properties and stability are inferior.

relation (23) for the reflection factor $\mathcal{R}_j$ and made a series of substitutions to eliminate the intermediate $\mathcal{R}_j$s. The result was a quotient for $\mathcal{R}_p$ with groups of terms in the numerator and denominator corresponding to 1, 3, 5 . . . . reflections. Schröder gave numerical examples showing the accuracy obtainable from the first and second approximations and from these it seems clear that for numerical work the method has little to commend it. However, the forms of the terms of different orders give useful information about the general run of the reflection factor curve. It may be possible to obtain an estimate of the number of films necessary to obtain an R–λ curve with given maximum R, a given number of maxima and minima or a given maximum gradient.

DESIGN METHODS

There is, of course, no synthetic design method which will enable a multilayer to be designed with specified properties. However, trial-&-error methods are used and also some devices for particular cases are known. Some of these will be described, necessarily in an unsystematic way, in the following sections.

Available Materials

Not many materials are available for vacuum deposition, the method of making thin films which is the most susceptible of precise control, and the optical constants of these are not known very accurately, partly because of the difficulty of making accurate measurements of optical constants on thin films, and partly because the optical properties seem to depend to a considerable extent on the conditions of evaporation. However, it happens conveniently that the properties of multilayers are generally much more critically dependent on optical thickness, which can be accurately controlled, than on refractive index and dispersion, so that the lack of accurate data is not as serious as it might be. In this section a short collection of values of refractive index and, in the case of metals, reflection factor for opaque layers is given. Further data are given in the references cited and also by Rouard (1952). No references are given for the data shown in the table

since it is not claimed that these data are critically chosen. A carefully compiled collection of thin film data would be very valuable, but it is doubtful whether evaporation conditions are sufficiently standardised to make this possible at present.

Symmetrical Multilayers

The matrix of a single film according to Equ. (19) has equal elements in the principal diagonal but, as can easily be seen by a few trials, the matrix of a multilayer does not in general have this property, so that a multilayer cannot be replaced by a single film of suitable thickness and refractive index*. If the multilayer is symmetrical, *i.e.* the thicknesses and refractive indices encountered are the same from either side, then it follows from Equ. (20) for the reciprocal matrix that the principal diagonal elements must be equal and so the symmetrical multilayer can be replaced by a single film of suitable effective thickness and index. The effective refractive index may be different from that of any known material and it will in general vary with wavelength fairly rapidly and in a different way from real refractive indices. In fact, both effective refractive index and thickness may turn out to be complex even if the multilayer is non-absorbing. Let a_{ij} be the matrix for the symmetrical multilayer; then, if g' and n' are the equivalent optical thickness and refractive index, it follows from Equ. (19) that

$$\left.\begin{aligned} \cos g' &= a_{11} = a_{22} \\ n' &= \frac{i \sin g'}{a_{12}} = \frac{-i a_{21}}{\sin g'} \end{aligned}\right\} \qquad (31)$$

This property of symmetrical multilayers was pointed out by Epstein (1952) who used it in designing filters. Epstein gave graphs of effective thickness and refractive index against relative wave-numbers (the abscissa is actually the optical thickness in phase units) for a number of three-component symmetrical multilayers having different ratios of thickness between the components and different refractive index arrangements. It can be seen from his graphs that in the real region the effective thickness is nearly equal to the total thickness of the multilayer but the effective refractive index varies rapidly over wide ranges.

Epstein used symmetrical multilayers of the form ABA as components of antireflection coatings : It is known that more efficient coatings, having two or more zeros of R in the spectrum, could be made if certain refractive indices were available and these Epstein simulated with ABA multilayers. The result was good but the spectral range was somewhat restricted because it is not possible to choose both index and thickness arbitrarily in an ABA combination; the thicknesses had to be so large that the complete coating behaved as if it was composed of films, thicknesses of which had the required values plus a half-wavelength, in the usual terminology second-order coatings. This is a general disadvantage of the method of symmetrical components, *i.e.* the design obtained may have greater thicknesses and consequently more rapid wavelength variation than a design obtained by, say, the approximate graphical method discussed on page 12. In some cases it is possible to circumvent this difficulty by a method which does not seem to have been suggested elsewhere. This is to make the total optical thickness of the basic ABA combination much smaller than $\lambda/4$ and to use several combinations in series to make up the required optical thickness. Let n_a and n_b be the component refractive indices and let $\rho = 2g_a/g_b$ be the ratio of the optical thicknesses, then, if g_a and g_b are less than $\lambda/8$ the equivalent refractive index can be shown to be approximately

$$n' = n_a \left\{ \frac{p+(n_b/n_a)}{p+(n_a/n_b)} \right\}^{\frac{1}{2}} \qquad (32)$$

and the equivalent thickness g' is approximately $2g_a+g_b$. It can be seen that n' must be between n_a and n_b, a restriction which does not operate in the general case, but the dispersion of n' is very small over a reasonable wave-number range.

Epstein's method can be used to determine the properties of periodic multilayers, since these can always be considered as of the form $(ABA)^m$ plus an

* It can be replaced by *two* films, as was shown by Herpin (1947).

extra film at one or the other end. The equivalent thickness g' and refractive index n' of the fundamental period ABA are found at each wavelength and then the multilayer $(ABA)^m$ is equivalent to a single layer of thickness mg' and refractive index n'. It is doubtful whether this leads to any saving of time in numerical computation but the concept is useful in design work. For example, in Fig. 7 are plotted the equivalent thickness and refractive index of the multilayer of which the reflection factor is given in Fig. 4; only real values of g' and n' are plotted. It may happen that it is desired to reduce the subsidiary maximum at $k=1.31$, *e.g.* for a dichroic beam splitter. It can be seen from Fig. 7 that in this region the multilayer has an equivalent refractive index of 2.7, so that an anti-reflection coating for this refractive index must be applied. If no suitable material is available the coating can be of the form ABA, chosen from Epstein's graphs. In this calculation the phase change on reflection (about 30°, Fig. 4) must be allowed for. This procedure can considerably reduce the unwanted high reflection but it is not completely effective because the effective refractive index changes so rapidly. This suggests building up the multilayer from a symmetrical period of which the effective refractive index is a slowly varying function of wavelength. For a period consisting of a quarter-wavelength of zinc sulphide sandwiched between two one-eighth wavelengths of magnesium fluoride it appears from Epstein's graphs that the effective refractive index is slowly varying and nearly unity on the long-wave number (short wavelength) side of the high reflecting zone. Thus a multilayer consisting of four or five such periods, *i.e.* the multilayer of Fig. 4 plus a $\lambda/8$ film of magnesium fluoride on either side, has the subsidiary reflection maxima considerably reduced, to around 5% in fact. (See long-chain line in upper part of Fig. 4, the ordinates beyond $k=1.6$ are less

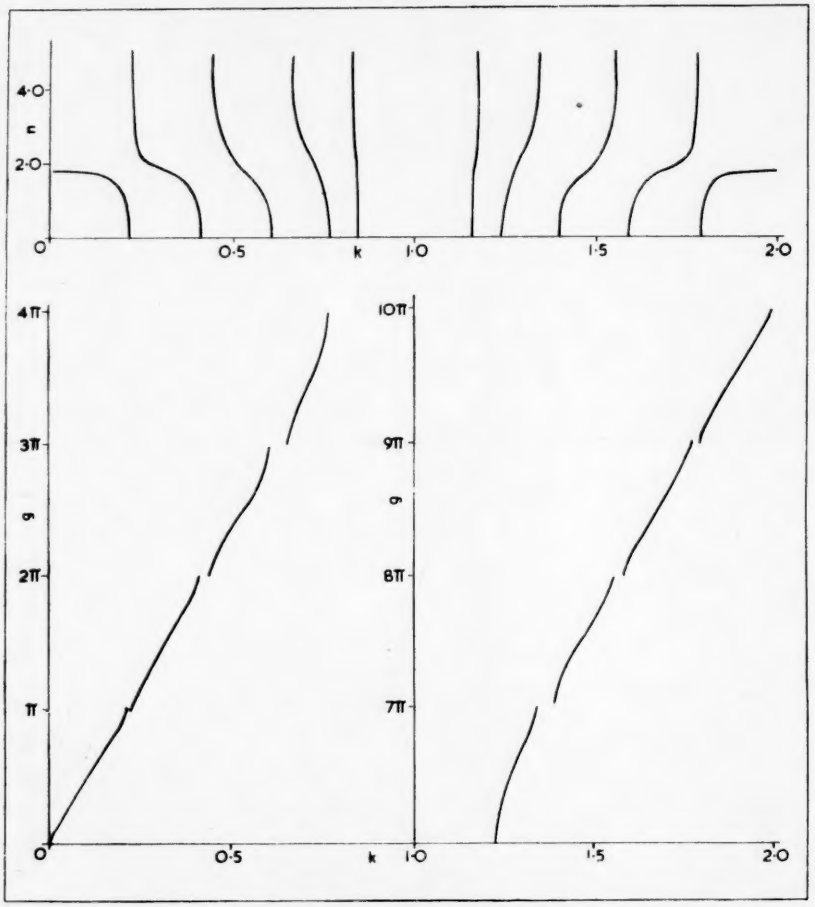


Fig. 7. Equivalent refractive index and optical thickness of high-reflecting multi-layer.

Vol. 4 1954

than 0.01). The maxima on the short wave-number side are correspondingly increased.

Similarly, if the fundamental period is $\lambda/4$ magnesium fluoride with $\lambda/8$ zinc sulphide on either side the short wave-number maxima are decreased. In this way quite satisfactory dichroic mirrors can be designed for normal incidence.

Reducing the Reflection Factor of a Multilayer

It sometimes happens that the reflection factor for an arbitrary multilayer, not necessarily symmetrical, must be minimised in a certain region. Here it sometimes helps to take a direct approach and see what can be done by adding a single film.

Let the matrix of the multilayer be b_{ij} and let it be immersed in media of generalised refractive indices u_1 and u_2, the light being incident from medium u_1. Suppose a single film of optical thickness g and index u is to be added between medium u_2 and the multilayer so as to annul the reflection. The condition for this is found to be :—

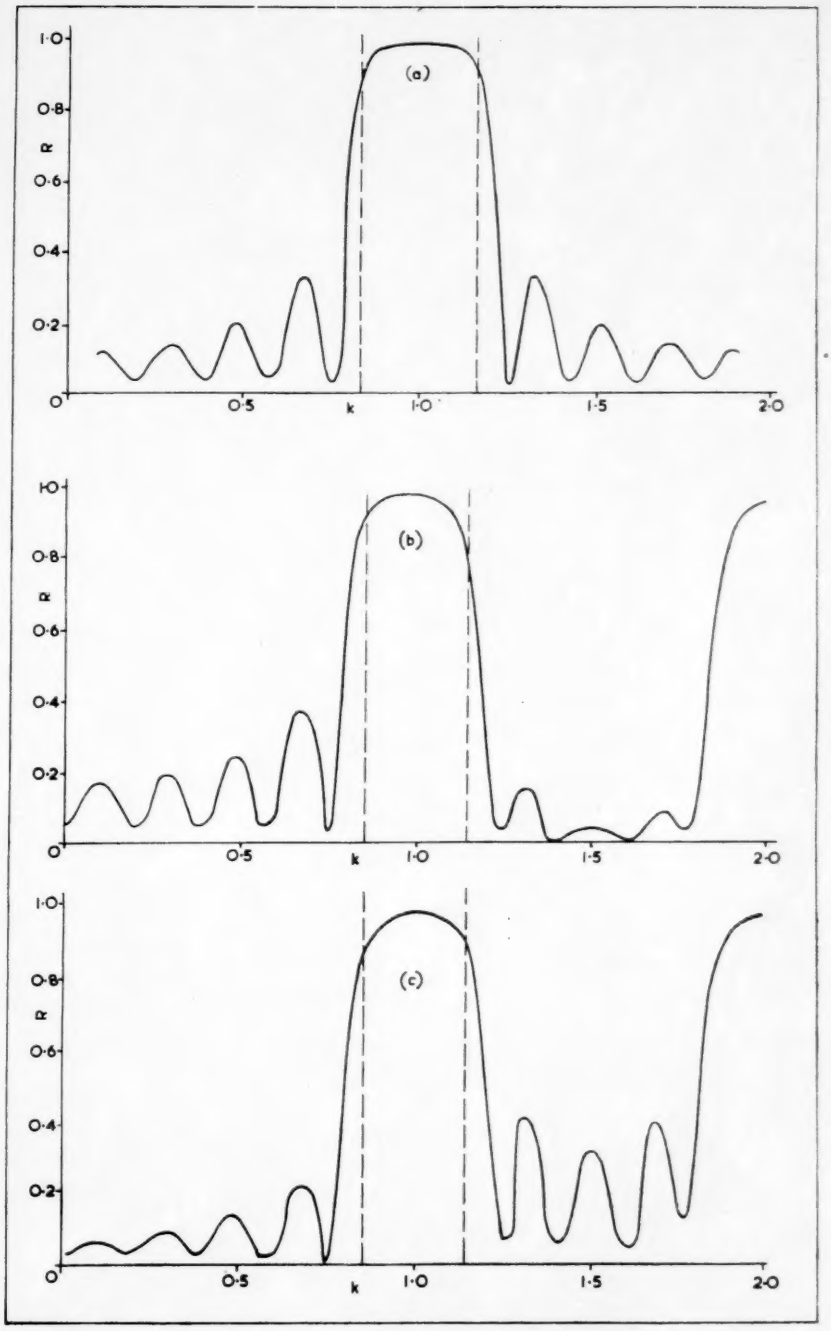


Fig. 8. Effect of varying thickness ratio in high-reflecting multi-layer.

$$(u_1 b_{11} + u_1 u_2 b_{12} - b_{21} - u_2 b_{22}) \cos g =$$
$$i\,(u b_{11} + u\,u_2 b_{12} - \frac{u_1}{u} b_{21} - \frac{u_1 u_2}{u} b_{22}) \sin g \qquad (33)$$

These two equations (two on separating real and imaginary parts) have to be solved for u and g; in the most general case this is all that can be done but if the multilayer is non-absorbing b_{21} and b_{22} are real and b_{12} and b_{21} pure imaginary and the solution can then be written as follows:

$$u^2 = \frac{u_1 \left\{ u_1 u_2 + b_{21}^2 - u_2^2 b_{22}^2 \right\}}{u_1 b_{11}^2 - u_1 u_2 b_{12}^2 - u_2} \qquad (34)$$

$$\tan g = \frac{u_2 b_{22} - u_1 b_{11}}{i \left(\frac{u_1 b_{21}}{u} - u u_2 b_{12} \right)}$$

$$= \frac{b_{21} - u_1 u_2 b_{12}}{i \left(\frac{u_1 u_2}{u} b_{22} - u b_{11} \right)} \qquad (35)$$

In practice u could take only a few fixed values and these would be substituted in Equations (35) to find the value of g which gave the nearest fit in both equations.

If this technique is applied to the problem of reducing the subsidiary reflection maxima in the passing zone of a high-reflecting multilayer, as in Fig. 4, the same result is obtained as in the previous section, *i.e.* $\lambda/8$ coatings must be used on each side.

Further Aspects of Beam-Splitter Design

Some further points can be illustrated by their application to the problem of dichroic beam splitters.

As indicated on page 12 the use of unequal thicknesses in the fundamental period of a high reflecting multilayer sometimes decreases the subsidiary reflection maxima. Fig. 8 shows a comparison between the reflection factors of three multilayers, (a) of equal thickness, (b) of high-index material thicker than (a) and (c) of low-index material thicker than (a). In each case there are 5 high-index and 4 low-index layers on the glass base. It can be seen that in (b) and (c) where the thickness ratio is 13 : 7 the subsidiary maxima on the long and short wave-number sides respectively are reduced. It may be conjectured that a combination of this principle with those discussed on pages 15 and 17 might yield very good beam-splitters, but this has not yet been tried, to the writer's knowledge.

When a dichroic beam splitter is to be used at oblique incidence the difference in properties with respect to the two polarisation directions causes difficulties. For example, with an angle of incidence in air of 45° the effective refractive indices of zinc sulphide and magnesium fluoride become 2.42 and 1.58 for the electric vector in the plane of incidence (say case (a)) and they are 2.19 and 1.15 for the other polarisation (case (b)). It then follows that in case (a) the high reflection zone extends over the relative wave-number range $0.8667 < k < 1.1333$ and in case (b) over $0.798 < k < 1.2014$. This discrepancy between the high reflecting zones for the two polarisations makes the division between reflected and transmitted wavelengths more gradual, which is in general undesirable in dichroic beam splitters. Some gain may be obtained by using higher refractive indices in both media, but here we are limited by available materials and by the fact that the width of the high reflecting zone decreases with decreasing disparity between the refractive indices. Also a slight gain can be obtained by varying the ratio of the thicknesses in the media; from the multilayer of Fig. 8(b) the upper limits of k for the two cases are 1.115 and 1.171, which are in slightly better accord. However, the best hope seems at present to lie in designing the rest of the apparatus so that angles of incidence less than 45° can be used. Fortunately it is often possible to do this at the cost of some increase in mechanical complexity.

CONCLUSIONS

In this paper some of the techniques in use for multilayer design have been outlined. The subject is developing rapidly and the writer does not claim to have treated it exhaustively. Future progress must depend for its stimulus on the solution of practical problems of durability and stability of multilayers

Vol. 4 1954

and accuracy of production.

It may be worthwhile to state here why the analogy with electrical filter network design has been little stressed : Filter networks are more complicated in that a chain of four-terminals, the analogue of thin films, is only one of the forms which they can take. They are also simpler in that there are no analogues to the polarisation and obliquity effects which occur in multilayers. Finally the dependence on frequency is usually quite different in the two cases. Thus filter network design techniques are quite different and there seems to be little of value in them for the multilayer designer, apart from the Smith chart.

Appendix on Matrices

The matrix algebra necessary for following the above paper may be explained in a few lines. A matrix expresses a linear transformation from one pair of variables E_1, H_1 to a second pair E_2, H_2.* The transformation is

$$\left.\begin{aligned} E_2 &= a_{11}E_1 + a_{12}H_1 \\ H_2 &= a_{21}E_1 + a_{22}H_1 \end{aligned}\right\} \qquad \text{(A1)}$$

or in matrix notation

$$\begin{pmatrix} E_2 \\ H_2 \end{pmatrix} = \begin{pmatrix} a_{11} & a_{12} \\ a_{21} & a_{22} \end{pmatrix} \begin{pmatrix} E_1 \\ H_1 \end{pmatrix} \qquad \text{(A2)}$$

A further transformation to the variables E_3, H_3 may be given by

$$\begin{pmatrix} E_3 \\ H_3 \end{pmatrix} = \begin{pmatrix} b_{11} & b_{12} \\ b_{21} & b_{22} \end{pmatrix} \begin{pmatrix} E_2 \\ H_2 \end{pmatrix} \qquad \text{(A3)}$$

It can be verified by simple algebra that the result of eliminating E_2 and H_2, *i.e.* applying both transformations in series, is

$$\begin{pmatrix} E_3 \\ H_3 \end{pmatrix} = \begin{pmatrix} b_{11}a_{11}+b_{12}a_{21} & b_{11}a_{12}+b_{12}a_{22} \\ b_{21}a_{11}+b_{22}a_{21} & b_{21}a_{12}+b_{22}a_{22} \end{pmatrix} \begin{pmatrix} E_1 \\ H_1 \end{pmatrix} \qquad \text{(A4)}$$

This shows the rule for multiplying together the b and a matrices, in that order, *i.e.*, to obtain the element in row i and column j of the product matrix take the scalar product of the ith row of the multiplier and the jth column of the multiplicand. The continued product of any number of matrices can now be formed by repeated applications of the rule. Obviously, this multiplication is not cummutative, *i.e.* $(a_{ij})\,(b_{kl}) \times (b_{kl})\,(a_{ij})$.

* In the most general case the relationship is between an original set of n variables and a related set of m variables, giving an $m \times n$ matrix: here we need only 2×2 matrices.

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OPTICAL SEMI-REFLECTORS

suitable methods and materials

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Summary

THE OPTICAL properties and methods of preparation of various materials—metals, semi-metals and dielectrics—are surveyed which may be used as optical semi-reflectors. Specific suggestions are made for obtaining any desired reflectance.

Sommaire

LES PROPRIETES optiques et méthodes de préparation de différents matériaux, métaux, semi-métaux et diélectriques qui peuvent être employés comme semi-réflecteurs optiques sont passés en revue. Des suggestions précises sont faites pour l'obtention de surfaces réfléchissantes quelconques.

Vol. 4 1954

INTRODUCTION

A WIDE RANGE of optical semi-reflectors has recently been prepared and examined by the authors, mainly in connection with the problem of injecting a graticule into the field of view of a telescope. Neither the problem itself nor the processes investigated were novel, but the information available is so scattered that a comparative review may be of value.

The films used may be divided into three broad classes : Metals, semi-metals and non-metals (dielectrics). The efficiency of the films (defined as the sum of the reflection and transmission factors) is lowest for the metals and highest for the dielectrics. The films were deposited on plate glass, mostly by vacuum evaporation, but in a few cases by sputtering. All the work was carried out in a coating unit with a 12-inch bell jar. Spectral transmission measurements were made with a Unicam SP600 photo-electric spectrophotometer.

* MS. received March, 1955.

METAL FILMS

Of the metallic films, silver gives the highest reflection, and also the highest efficiency, in the visible part of the spectrum. For a semi-reflector having equal reflection R and transmission T the efficiency is about 80%. The metal is easy to deposit by evaporation from a molybdenum or tantalum boat, but does not adhere well to glass and is readily tarnished unless protected. Nevertheless, it is widely used in multiple-beam interferometers, where it has been challenged only recently by dielectric materials which in the form of multilayer films have a lower absorption[1].

Aluminium is widely used as a semi-reflector, as it is easy to evaporate, adheres strongly to glass and is reasonably neutral in both transmission and reflection. In addition the metal is comparatively durable without further protection on account of the natural oxide layer which rapidly forms in air. For equal transmission and reflection the efficiency is about 70%.

Because of their general utility aluminium films were studied with a view to obtaining semi-reflectors of known properties without photometric monitoring. It was found that sufficient reproducibility for most practical purposes could be obtained by evaporation of a known mass (*i.e.*, a known length) of wire from a horizontal tungsten filament at a given distance below the glass plate[2]. To ensure consistency, a blank evaporation was carried out when a new filament was brought into use.

Fig. 1 shows the transmission as a function of mass of aluminium, with the glass 15 cm. above the filament. For other distances, the mass of aluminium required may be calculated using the inverse square law, bearing in mind that for short distances coatings will be noticeably thinner near the edge than the centre[3]. Fig. 2 shows the variation of the reflection factor and efficiency with transmission.

While it is easy to obtain reasonably good films, the best films are obtainable only within a narrow range of conditions[4]. Adherence is best, and blistering is prevented, by raising the temperature of the glass to 100°C. If the glass is above 120°C, the film is deposited with a bluish ' bloom '. The pressure in the chamber should be below 10^{-4} mm. Hg, otherwise the reflectivity in the violet (and ultra-violet) falls off and the absorption increases. At higher pressures it is also impossible to obtain consistency of deposition by evaporation of a given weight of metal. The evaporation rate affects adhesion, and for a distance of 15 cm. between filament and source, the optimum rate is about 10 mg. in one half-minute.

Chromium films are sometimes useful, on account of their hardness and resistance to chemical attack, and may readily be deposited by sublimation from a tantalum boat. Rhodium has an even higher physical and chemical resistance to attack, and rhodium semi-reflectors are virtually indestructible, as well as being almost neutral in colour. The films are prepared by evaporation from an electroplated tungsten filament but to secure the best adhesion to glass the use of a mercury, rather than an oil, diffusion pump is usually advocated[5]. The efficiency of chromium and rhodium films is, unfortunately, low.

The spectral transmission characteristics of the

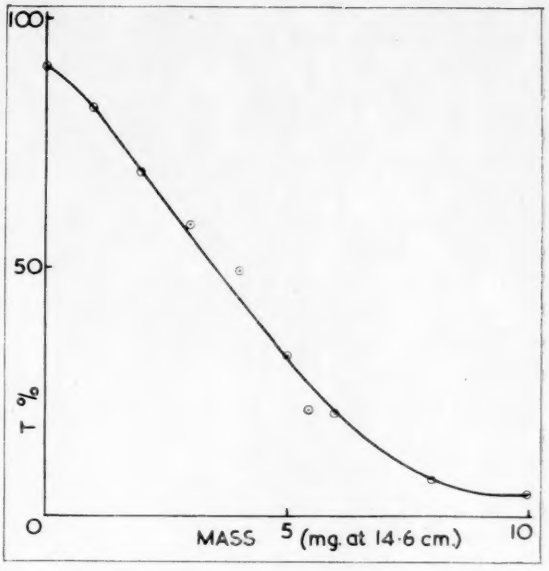

Fig. 1. Variation of transmission with mass of aluminium evaporated.

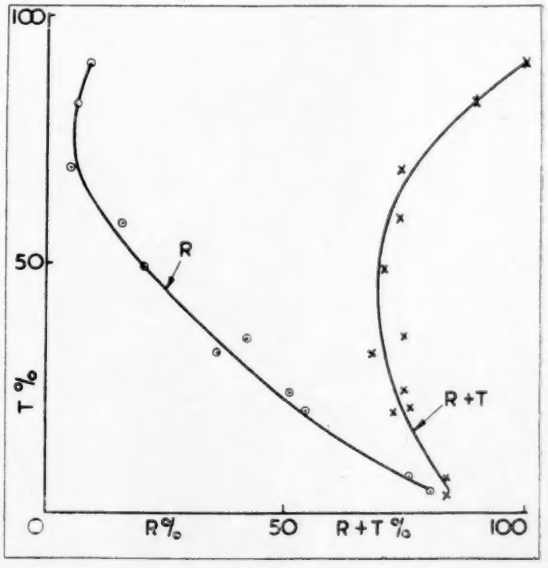

Fig. 2. Variation of R and of $R+T$ with T for aluminium on glass.

various metals are of interest, particularly in connection with the preparation of neutral filters. Measured values are shown in Fig. 3 where the experimental points are omitted for reasons of clarity. The ordinate is optical density. As with aluminium, the shapes of the curves depend to some extent on pressure, those here all being appropriate to 10^{-4} mm. Hg or less. If an alloy of 80% nickel and 20% chromium is evaporated at low pressure, a film almost neutral throughout the visible spectrum is produced[6]. At 10^{-3} mm. Hg or higher, practically pure chromium is deposited, with a corresponding change in transmission.

Changes in thickness of the films cause changes in the shapes of the plotted curves. For example, if the thickness of a gold film is doubled, the density in every part of the spectrum will (to a reasonable approximation) also be doubled, so that the dip in the blue-green is accentuated.

SEMI-METAL FILMS

Optically, the semi-metals are characterised by having high values of the refractive index n, but low values of the extinction co-efficient k. Under certain conditions it is therefore possible to produce films having a reflectivity and absorption intermediate between those of the metals and the dielectrics. With silicon[7], the type of film obtained depends on the vacuum conditions. With slow deposition in a good vacuum, 10^{-5} mm. Hg, $n=4.2$, $k=0.10$ and the highest reflectivity theoretically attainable is $R=54\%$ with the corresponding transmission $T=24\%$. A film approaching the theoretical optimum was prepared, with $R+T=78\%$. The efficiency is thus only slightly higher than that for aluminium, but silicon appears to be appreciably tougher. Molten silicon attacks most boat materials and the films are thus rather difficult to prepare. It was, however, found possible to prepare one film with each tungsten foil boat. Alternatives are the use of beryllium oxide crucible[7], or a massive tungsten boat if sufficient filament current can be provided[8].

Other semi-metals investigated included germanium and tellurium. They appear to be of little value as semi-reflectors, as both are soft and of low efficiency. Also they are not neutral in colour. Germanium is a somewhat capricious material to evaporate and gives films with a white light efficiency of about 66%, at $R=40\%$. With tellurium, the corresponding efficiency is only 50%.

The spectral transmission curves of silicon, germanium and tellurium films having nearly maximum reflectivity are shown in Fig. 4.

DIELECTRIC FILMS

Where only a moderate increase in reflectivity compared with plain glass is required, single-layer dielectrics are suitable because many of them will give films of very high efficiency. The reflection and transmission properties of all dielectric films are strongly influenced by interference effects. With single-layer films the reflectivity is highest when the optical thickness (*i.e.* actual thickness x refractive index) is $\lambda/4$, so that constructive interference takes place for the wavelength λ.

Zinc sulphide ($n=2.35$) was one of the first materials to be used[9] and enables a reflectivity of about 30% to be obtained. The layer is neutral in colour because the interference maximum is quite broad; it has an efficiency of 99% or better and is moderately durable. The durability may be increased by pre-heating the glass substrate to as high a temperature as will allow the sulphide to condense.

The only practical difficulty concerning the evaporation, is a tendency for the sulphide to spatter, but this may be overcome by wrapping the tantalum foil closely with a tungsten spiral. The maximum reflectivity may be judged fairly precisely by direct inspection during the evaporation. A more sensitive method is to use a monitor plate, already coated for maximum reflectivity, and then evaporate until it shows a minimum (purple colour).

Antimony tri-sulphide is very attractive at first sight, on account of its high refractive index ($n=3.2$). It is readily evaporated, but unfortunately the efficiency is rather low, as would be suspected from the colour of the bulk powder, the film being brown-

Vol 4 195

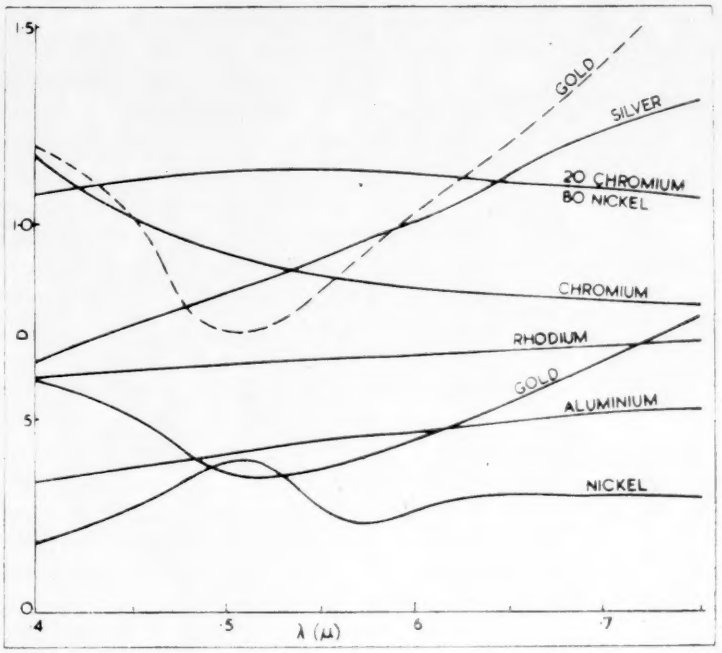

Fig. 3. Variation of transmission density with wavelength for some metals.

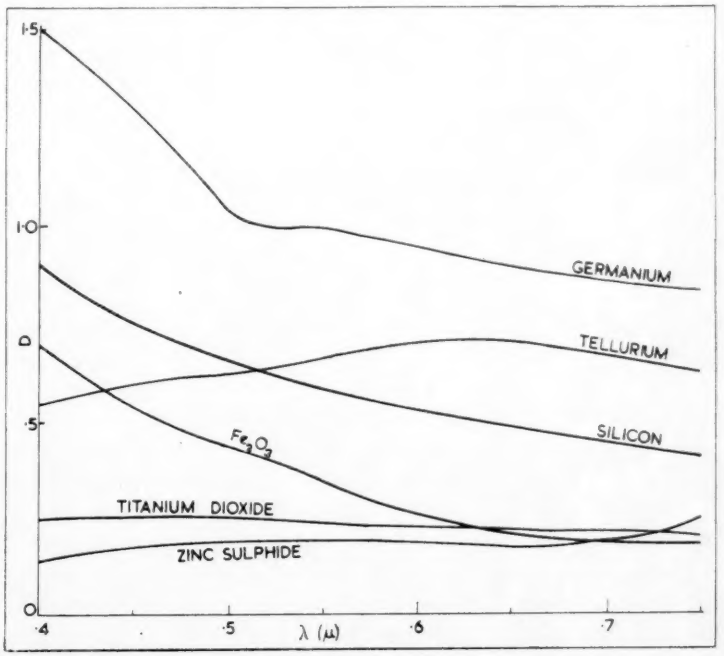

Fig. 4. Variation of transmission density with wavelength for some semi-metals and dielectrics.

ish. The highest reflectivity obtainable is 38%, the corresponding transmission being 51% (*i.e.*, $R+T=$ 89%). The films are also rather soft and fragile, although some increase in toughness may be obtained by pre-heating and subsequent baking of the glass.

Lead sulphide, with an even higher refractive index $n=3.9$), gives films which are still softer and less efficient. For maximum reflectivity, $R=37\%$, $T=$ 25% and $R+T=62\%$. For a reflectivity of 20% the efficiency is about 75%. It is thus of no practical value.

Table 1. **Optical and performance data of various materials.**

Material		*Max. Reflectance %*	*Efficiency %*	*Remarks*
Silver	Ag	94	80*	Tarnishes easily.
Aluminium	Al	88	70*	Readily applied.
Rhodium	Rh	76	62*	Tough, neutral.
Silicon	Si	54	78	Tough.
Germanium	Ge	45	66	Soft, reddish.
Tellurium	Te	40	50	Soft.
Zinc Sulphide	ZnS	30	99	Neutral.
Antimony Sulphide	Sb_2S_3	38	89	Soft, reddish.
Lead Sulphide	PbS	37	62	Soft, greyish.
Titanium Oxide	TiO_2	42	99	Tough, neutral.
Ferrous Oxide	Fe_2O_3	52	88	Tough, reddish.
Bismuth Oxide	Bi_2O_3	38	96	Neutral.
Silicon Monoxide	SiO	20	96	Tough, neutral.

* Efficiency quoted for $T = R$.

Films of titanium oxide may be prepared in several ways[10]. Evaporating the oxide directly leads to decomposition, so that the resultant films consist of a mixture of Ti and TiO_2, with a low optical efficiency. It was found more satisfactory to evaporate pure titanium metal, in sponge form, from a tungsten wire spiral, closed at the bottom with a piece of tantalum foil. In this way, a film of titanium metal is formed, which should have a transmission between 5 and 10%, the higher value yielding films with the highest ultimate transmission and efficiency. The metal film must then be baked in air for 2 or 3 hours at 400°C to oxidise it completely. Small pieces of glass may be baked quite successfully on a hotplate which is shielded at the sides but left open at the top. Complete enclosure of the glass tends to cause the final oxide film to be spotty or patchy. The temperature of the glass should be increased or decreased slowly over a period of several hours. With care, films having a reflectivity close to 42%, the theoretical maximum (corresponding to a refractive index of 2.7) may be obtained with an absorption loss of only 1%. The films are almost neutral in colour, as is shown in Fig. 4, and are extremely durable.

Iron (ferrous) oxide films may also be prepared by evaporation of iron, followed by subsequent baking in air[11]. The evaporation is rather difficult to perform as molten iron alloys with the normal boat materials. It may be evaporated from a beryllium oxide crucible. It was also found possible to evaporate directly from a spiral of pure iron wire, but the margin between the evaporating temperature (about 1,400°C) and the melting point (1,530°C) is small. In order to obtain the highest ultimate reflectivity, the evaporation of the iron must be continued until the transmission is 16-17%. This value is rather critical and, in the absence of photometric monitoring equipment, the correct value may be obtained by comparison of the specimen under preparation with layers of known transmission. Subsequent baking is then carried out as described for titanium. A typical specimen prepared with an initial transmission of about 20%, had a reflectivity of 36%, with an efficiency of 92%. A German sample (produced during the late war) had equal reflectance and transmission of about 45%. The highest reflectance theoretically obtainable is 52%, with about 12% absorption. The films are remarkably tough, but are reddish by transmission. Great care is required to prepare a film which is free from blemishes.

Bismuth oxide films were produced by evaporation of bismuth, followed by baking in air. With an initial transmission of 20%, the most efficient final plate had a reflectance of 15% with an efficiency of

Vol. 4 1954

90%. A more satisfactory method of preparation appears to be the sputtering of bismuth[12] in an atmosphere of argon and oxygen. This enables a controllable layer of the oxide to be deposited directly. The oxygen content is quite critical, as a deficiency leads to a mixture of bismuth and its oxide being deposited, with attendant high absorption; an excess leads to a lowering of the refractive index with consequent loss of reflectance. Cadmium oxide films may be prepared in a similar manner by sputtering cadmium, but they have little merit purely as semi-reflectors. Both cadmium and barium oxide films produced in this way are soluble in dilute acids.

Remarkably tough films of silicon monoxide* may be made by direct evaporation from a tungsten spiral closed at the bottom with tantalum foil. A reflectance of about 20% was attained with an efficiency in excess of 96%. The reflectance and efficiency obtainable appear to depend on the source of supply, as the American powder appears more white than the British. The refractive index reaches its maximum value (1.97) with rapid evaporation under a good vacuum[13].

The spectral transmission characteristics of some of the semi-metals and dielectrics are shown in Fig. 4. All the curves are appropriate to the layer which has the highest reflectivity over the visible spectrum as a whole. Increase or decrease of thickness of the layers will reduce the transmission density, because of the dominating importance of interference effects.

SUMMARY AND CONCLUSIONS

The Table shown on the opposite page summarises some of the information of the preceding sections in order to enable a choice to be made.

It may be concluded that, for a reflectance of 20%, silicon monoxide is the most suitable material; for 30%, zinc sulphide and for 40%, titanium dioxide. For reflectances over 40%, aluminium is probably the most satisfactory material for general use. Where a higher efficiency is required, multi-layer dielectrics such as alternate quarter-wave layers of magnesium fluoride and titanium dioxide[10] appear to be a better proposition than single layers of silicon or ferrous oxide, which in any case can take the reflectance to about 50% only.

This paper is published by permission of the Chief Scientist, Ministry of Supply. Acknowledgement is also made to the controller, H.M.S.O., for permission to publish this paper.

* Silicon monoxide may be prepared by roasting *in vacuo* an equimolecular mixture of silicon and silica (the dioxide). The monoxide is available commercially.

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SOME 'BLACK' INTERFERENCE REFLECTORS

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Summary

THE CONSTRUCTION of various types of interference mirror, designed to reflect near infra-red and to absorb visible radiation, is described. The spectral discrimination of the simple type of interference mirror may be enhanced by taking advantage of the optical properties of gold or germanium.

Sommaire

LA CONSTRUCTION DE TYPES variés de mirroirs à interférence, faits pour réfléchir aux environs des rayons infra-rouge, et pour absorber les radiations visibles est décrite. La discrimination spectrale de type simple de mirroir à interférence peut être rehaussée en prenant l'avantage des propriétés optiques de l'or ou du germanium.

Vol. 4 1954

INTRODUCTION

INTERFERENCE FILTERS, in which the desired spectral region is transmitted, and interference mirrors, in which the desired energy is reflected, have received much experimental and theoretical attention in recent years[1]. A number of filters of the second type have been prepared by the authors, to reflect near infra-red radiation and to absorb in the visible region. A cut-off as sharp as possible at about 0.8 micron was desired, and obtained for some mirrors, but others were prepared which discriminated in other regions of the visible spectrum. The interference coatings were intended for application to mirrors of large diameter, so that not more than two or three layers were permissible, of which the outermost at least had to be physically and chemically resistant.

TYPES OF MIRROR PREPARED

The simplest type of interference reflector consists of a full reflector, on top of which is deposited a spacer layer, followed by a partial reflector. An example of this type has already been described[2]. The base is aluminium, and the spacer layer is formed by anodisation at a suitable voltage to form a film of aluminium oxide of the correct thickness. Finally a thin layer of aluminium is applied. The specular reflection characteristics of two mirrors made in this way, shown in Fig. 1, reveal rather poor spectral discrimination.

It is possible to enhance the discrimination by making one of the layers of a material, the optical properties of which (refractive index or extinction coefficient) vary with wavelength in the required region. Substances which were considered in this connection were gold, germanium, tellurium and selenium. It was found that the absorption edge of tellurium is too far in the infra-red to be useful while that of selenium, although extremely sharp, depends too strongly on conditions of deposition, and on ambient temperature. Some reflectors consisting of a thin layer of selenium on aluminium showed good discrimination, but quickly deteriorated by crazing.

The spectral transmission[3] and reflectance of gold vary with wavelength and an opaque gold film shows a steeper cut-off than a simple interference reflector, as shown in Fig. 2. Enhanced reflection from metals is obtained by superimposing a quarter-wave film of low refractive index followed by a similar film of high refractive index[4]. Also, the minimum reflectance of the interference reflector may be made truly zero[1].

* MS. received March, 1955.

Thus by correct choice of the spacer layer and reflecting top layer, mirrors on a gold base may be made to have sharp discrimination with a low minimum and high maximum. This is illustrated in Fig. 2 which shows the spectral characteristics of three mirrors with magnesium fluoride spacers and aluminium partial reflectors.

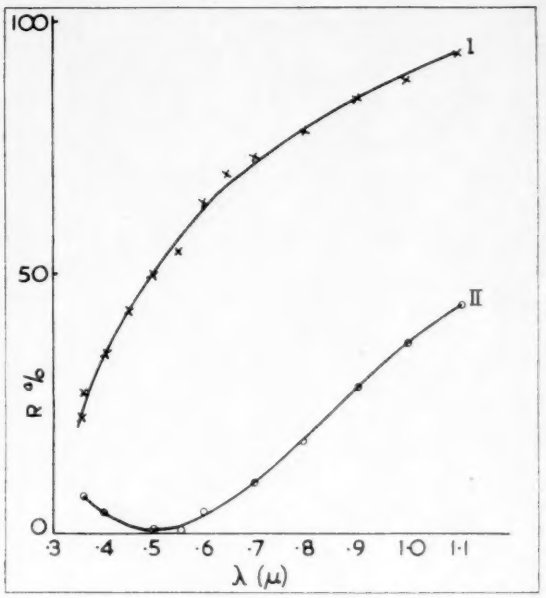
Filters incorporating gold are more tedious to make than the simple type as an additional evaporation process is required. Also, evaporated gold adheres rather badly to glass and still worse to aluminium. Strongly adhering layers of gold can, however, be applied to aluminium electrolytically. The spectral reflection characteristics of the simple and the gold mirrors vary markedly with angle of incidence of the radiation.

Further experiments were made with aluminium-base mirrors to reduce the required number of evaporations to two. Germanium, which adheres much more strongly than gold to aluminium but which is more difficult to evaporate, was used as the spacer and a dielectric (silicon monoxide) as the partial reflector. The spectral reflectance curves for two such mirrors are shown in Fig. 3. By suitable choice of thickness of the germanium, almost zero reflectance can be obtained over much of the visible spectrum. This is because the refractive index varies with wavelength in such a manner as to keep the optical thickness nearly constant. Of course the changing index affects also the reflected amplitudes at the metal-germanium, and the germanium-dielectric surfaces. This type of mirror has also been briefly described elsewhere[5].

The silicon monoxide layer is extremely tough, so that a very serviceable mirror results. The thickness of the coatings on a concave mirror may be graded so that the effective colour is uniform across the aperture when fully flashed. The gradation required is small because, owing to the high refractive index, light travels nearly normally in germanium even for large angles of incidence at the surface.

EXPERIMENTAL METHODS

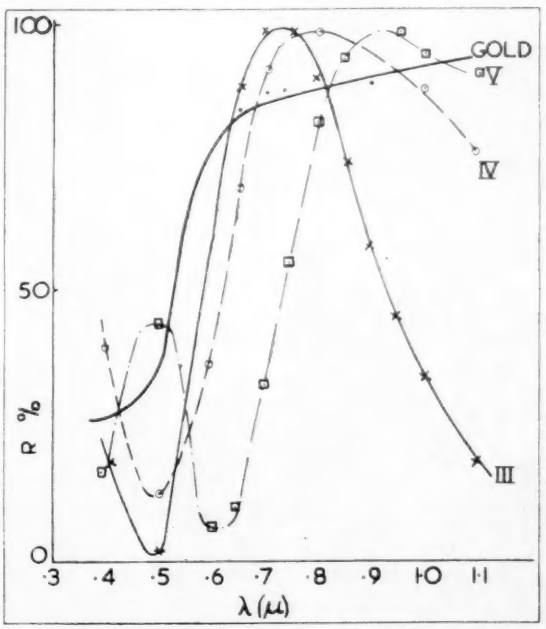
The simple interference reflectors were made in



Spectral reflectance curves of interference mirrors.

Fig. 1. (above): Al–Al_2O_3–Al mirrors.

Fig. 2. (below): Au–Mg F_2–Al mirrors.



accordance with methods already described[2]. Later, experiments were made with solid aluminium mirrors but initially, glass coated with two layers of evaporated aluminium was used. In this way freedom from pinholes which tend to enlarge on anodising was obtained. The mirror was made the anode of an electrolytic bath, with pure aluminium as the cathode. The electrolyte consisted of a 3% solution of tartaric acid to which was added sufficient ammonia solution to make the pH about 5.5. Methyl orange was found to be a suitable indicator. The initial current was limited to about 1 A/cm.^{2} but even so there was a tendency for the aluminium film to be loosened at the surface of the electrolyte, thus destroying the path of the current. This difficulty, which does not arise with a solid mirror, was overcome by waxing the aluminium in this region. The initial current falls rapidly and after about a minute the thickness of the oxide depends mainly on the applied voltage, 100 V corresponding to a thickness of about 1,300Å[2]. In the present experiments 35-37 V gave a spacer layer of correct thickness for 40° incidence. It was found necessary to use a battery as the source of e.m.f. since using d.c. mains sufficiently precise control could not be obtained possibly due to ripple. In initial experiments the top layer of aluminium was deposited at such a thickness[3] that the transmittance was about twice the reflectance. The thickness for mirrors prepared subsequently was adjusted to give a minimum in the desired spectral region.

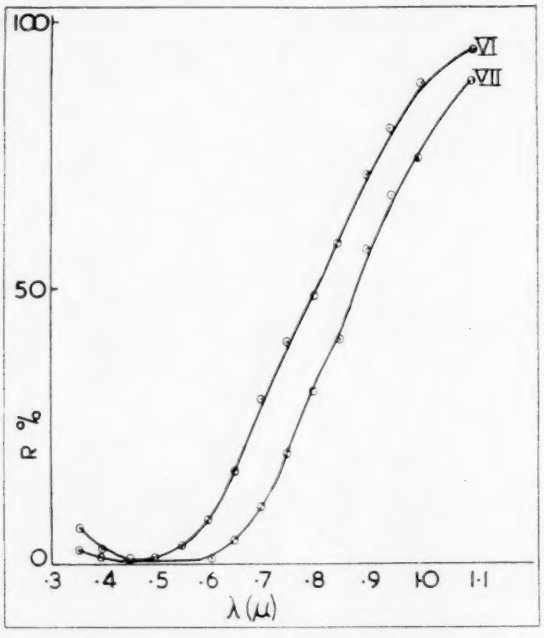


Fig. 3. Spectral reflectance curves of Al–Ge–SiO mirrors.

The second class of mirrors was prepared by evaporating a thick layer of gold on to glass, followed by an approximately quarter-wave thickness of magnesium fluoride. The thickness of the fluoride was monitored by observing the colour of the layer on top of a plain piece of glass adjacent to the gold mirror. The colours of the monitor were yellow, yellow-purple and purple for mirrors III, IV and V, final reflectances of which are shown in Fig. 2. The top layer, aluminium, was applied by evaporation until the desired minimum was secured. Other semi-reflectors[3] may be used but were not tried.

Several mirrors of the third type were made with germanium spacer layers of varying thicknesses. The colours at this stage, when viewed at 40°, varied from pale yellow to bright blue, corresponding to mirrors VI and VII of Fig. 3. To obtain extinction over as wide a region as possible, the germanium should have its highest reflectance at about 0.45 micron. The addition of the final semi-reflecting layer of the correct thickness then gives a minimum at this wavelength. For the top layer, silicon monoxide can be used as it has a suitable refractive index. It is also very tough but other semi-reflectors could be used. Germanium is somewhat capricious to evaporate, in that, for a given temperature, the rate of deposition on the glass may vary from very slow to unmanageably fast.

OPTICAL MEASUREMENTS

Measurements of absolute specular, spectral reflect-

Vol. 4 1954

ance were made with a Unicam S.P.600 photo-electric spectrophotometer. This was modified by removing the normal lamphouse and substituting an optical system consisting of a lamp, the mirror under test (held in a vertical plane) and a condenser. The lamp and specimen were mounted on a bench which could rotate about a vertical axis passing accurately through the reflecting surface of the specimen. The condenser was fixed and an image of the lamp was focused on to the position occupied by the original lamp. The specimen was adjusted until the required angle of incidence was obtained and the entrance slit was then imaged with light either from the lamp direct or *via* reflection at the specimen. The components were adjusted until in either position light from the same portion of the source, and leaving it in the same direction, was imaged on the slit and completely filled the collimating mirror in the spectrometer.

This paper is published by permission of the Chief Scientist, Ministry of Supply. Acknowledgement is also made to the Controller, H.M.S.O., for permission to publish this paper.

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954

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some theoretical and experimental observations on THE ULTIMATE VACUUM OBTAINABLE IN VAPOUR PUMPS

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Summary

A CRITICISM is advanced of the assertion still current, that the ultimate vacuum of a vapour pump is limited by the diffusion of gas back through the vapour blast. With an experimental device it has been shown that the ultimate vacuum with hydrogen is much lower than that resulting from backward diffusion. The occurrence of the gas escaping back through the vapour molecules, particularly in the case of hydrogen, has been proved and it has been shown that vapour condensation is not required for pumping action. It has been confirmed that the ultimate vacuum is limited by the presence of gases in the vapour stream from the boiler. From curves showing the variation of the ultimate vacuum with the fore pressure it is possible to distinguish between the prevalence of the gas escaping backward and the gas contained in the vapour.

Sommaire

UNE CRITIQUE est faite sur l'affirmation encore courante, disant que le vide maximum obtenu par une pompe à vapeurs quelconques est limité par la présence de gaz remontant au travers du jet de vapeur. Au moyen d'un dispositif expérimental il a été demontré que le vide maximum avec de l'hydrogène est plus bas que celui résultant de la pression du gaz remonté. Le fait que le gaz repasse au travers des molécules de la vapeur diffuse, particulièrement dans le cas de l'hydrogène, a été prouvé, et il a été demontré que la condensation de vapeur n'est pas nécessaire pour l'action de pompage. Il a été confirmé que le vide maximum est limité par la présence de gaz dans le jet de vapeur venant de la chaudière. Partant des courbes montrant la variation du vide maximum en fonction de la pression à la pompe primaire il est possible de distinguer la prévalence du gaz remontant au travers de la vapeur et le gaz contenu dans la vapeur.

Vol
4
195

INTRODUCTION

IN NUMEROUS PAPERS on the vapour pump in recent years, interest has been centred on its working principle, as well as on the factors limiting the lowest pressure obtainable with such an instrument.

Investigations by several workers have made it evident that there are some important considerations to be taken into account in order to eliminate the assertions which directly contradict the experimental measurements, nevertheless there still persist many contradictory views, mainly because of lack of reliable experimental results as well as incorrect theoretical ideas.

The general approach to the study of the working of the vapour pump should be to analyse the various phenomena which occur in its normal operation. Measurements on a commercial design do not give any conclusive results, mainly because the work cannot be carried on in all circumstances. At the present stage it is difficult to interpret the relationship between theory and experimental results on the vapour pump, because measurements involve many different processes, the results of which are very complicated. It is well known that measurements of pumping speed

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with hydrogen on various pump designs have led to a controversy.

Therefore we preferred to carry out some measurements on an experimental model. The aim was not to discuss in detail current practices in vapour pump design and construction but rather to examine some of the fundamental characteristics of the pump as well as the limitations of its performance.

It will become apparent as we proceed, that any serious study of the working of the vapour pump must be accompanied by an analysis of the behaviour of the vapour towards different gases in various conditions. So we will restrict our interest to investigations on oil vapour, the high molecular mass of which makes it suitable for pumping almost all gases and vapours.

In this paper an effort has been made to describe some convincing facts proved by experiment.

Vol. 4 1954

THEORETICAL CONSIDERATIONS

A brief historical review of the generally accepted diffusion theory has been given by the author in another paper[1]. Its originator obtained a formula which expresses the ultimate vacuum by supposing that there is equilibrium between the gas molecules entrained by the vapour blast and those diffused in the opposite direction along the pump casing. Supposing an arrangement, in which such an occurrence would take place, the mathematical description can be presented as follows.

Consider an intermediate isobar and let U be the speed of the vapour blast; the speed of the entrained gas will be aU, where $a<1$. Because of the pressure gradient the gas will diffuse in the opposite direction with a speed, which may be expressed by

$$-\frac{D}{n}\frac{dn}{dx}$$

where n is the molecular gaseous concentration at the level of the considered isobar and D is the diffusion coefficient.

Thus the effective speed of the gas streaming will be

$$V = aU - \frac{D}{n}\frac{dn}{dx} \qquad (1)$$

If the pressure distribution along the pump casing is uniform and the range of the vapour pressure is small, the above equation may be integrated to:

$$\frac{n_o}{n_f} = \exp\left(-\frac{aU - V}{D}L\right) \qquad (2)$$

n_o and n_f being the molecular gas concentrations at the two ends of the pump of length L.

In the steady state, in every isobar the entrainment speed of the gas is compensated by the speed in the opposite direction resulting from back diffusion. Then $V=0$, and the Equ. (2) becomes Matricon's[2] formula :

$$\frac{n_o}{n_f} = \frac{p_o}{p_f} = \exp\left(-\frac{NUL}{D_o}\right) \qquad (3)$$

where $D_o=ND$, N is the vapour molecular concentration and a is taken as unity.

The mathematical expression of the ultimate vacuum, Equ. (3), which asserts that the lowest pressure obtainable with a vapour pump is actually the gaseous pressure in the front of the nozzle resulting from the backward diffusion of the gas from the fore pressure side, could not be definitively demonstrated by experiment, although it was by a direct application of this result that Gaede had been guided to design his pump.

From the preceding formula the main conclusion is that the ultimate pressure p_o would be higher for lighter gases because D_o is larger.

However, considering the exhaustion process to express the pressure as a function of the pumping speed, a contradictory result will be obtained. The amount of gas per second flowing out of the vessel of volume V is given by :

$$q = p\frac{dV}{dt} = pS$$

where S is the pumping speed, supposing that the impedance of the connecting line between the vessel and the pump is negligible.

It is obvious that

$$pdV = -Vdp$$

and therefore

$$\frac{dp}{p} = -\frac{S}{V}dt$$

As the diffusion theory states that the pumping speed of a vapour pump is constant and equal to $S=cA/4$,

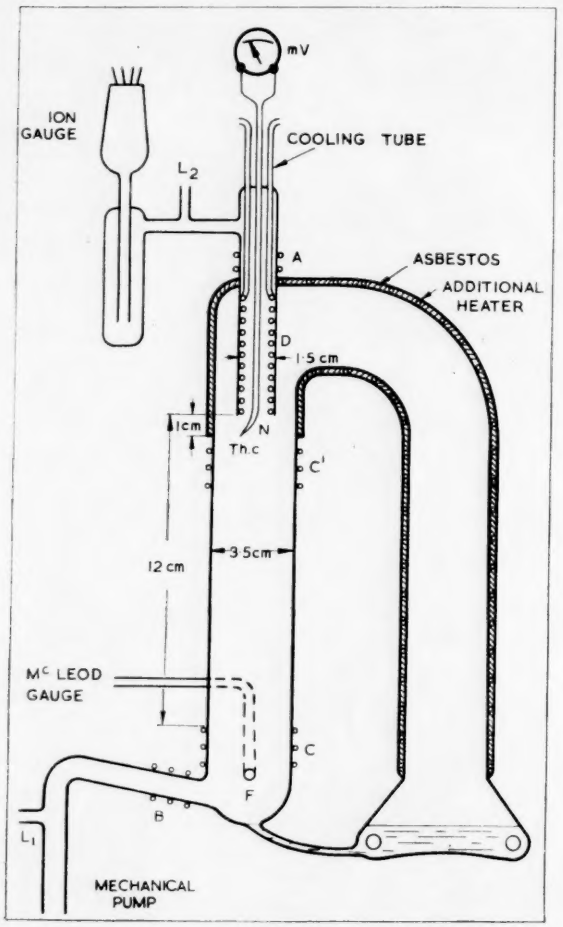

Fig. 1. Sketch of the experimental pump.

where c is the mean velocity of the gas molecules and A is the area of the pump aperture, the above equation may be integrated to

$$p = p_1 \exp\left(-\frac{cAt}{4V}\right) \qquad (4)$$

This equation gives the pressure p in the vessel at time t, if at time $t=0$ the pressure was p_1.

In practice, a given pump attains a limiting pressure after a time, depending on the magnitude of the vessel. This limiting value is imposed by the amount of leaking gas, *i.e.* gas desorbed from the walls and from the working fluid, as well as by the ultimate vacuum of the pump. Equ. (4) shows that for a lighter gas the pressure p will be lower than for a heavier gas after the same pumping time, because of the mean molecular velocity c.

Suppose that the volume V be very small, *i.e.* that the inlet of the pump be closed. The pressure at the inlet will first decrease rapidly with time. However, because of the unavoidable evolution of gas the pressure approaches asymptotically the lowest attainable value, thus reaches the steady state.

From Equation (4) it may then be concluded that the ultimate pressure would be lower for a lighter gas, a result which contradicts the fundamental conclusion of the diffusion theory, as stated in Equation (3).

On the basis of suitable assumptions the writer worked out a new theory of the vapour pump[1,3] which gives expressions for both the ultimate pressure and the pumping speed and whose conclusion agrees with the statement in Equ. (4).

The working principle of the vapour pump may be described as follows : In the boiler of the vapour pump, oil or mercury vapour is produced at a pressure up to several mm. Hg. This vapour flows through the jet tube and leaves the nozzle at high velocity, creating in front of it a very low partial gas pressure, by displacing the gas molecules over a gradient of increasing molecular concentration, towards the next nozzle or the fore pressure outlet. Such a gradient concentration is maintained by the impact of the vapour molecules with the gas molecules. Gas molecules diffusing into the pump are continuously entrained by the vapour jet. Before reaching the outlet the vapour condenses and flows back to the boiler.

If n_o is the gas concentration at the level of the nozzle, and n_f the concentration at the fore pressure outlet, the ratio :

$$r = \frac{n_o}{n_f} = \frac{p_o}{p_f}$$

expresses the vacuum efficiency of the pump.

The mathematical expression of the ratio r may be determined by considering the steady state along the pump casing due to the equilibrium between the forces of impact of the vapour molecules and the thermal agitation of the gas molecules. The analytical operation has been worked out in another paper[1], by equating the momentum of the gas pressure gradient given up per unit section to that transmitted by the vapour impact. This resulted in the following formula for the ultimate pressure obtainable with an ideal vapour pump :

$$p_o = p_f \exp\left(-\frac{3\sqrt{\pi}}{2}\,\alpha\rho^2 N_v\,\frac{m_v c_g U}{m_g v_g^2}\,\frac{\psi_a}{a}\mathrm{L}\right) \quad (5)$$

where

N_v = vapour concentration,
m_v, m_g = the masses of vapour and gas molecules respectively,
U = the speed of the vapour blast,
v_g = the square root of the mean square speed of the gas molecules,
c_g = the most probable speed of the gas molecules,
ρ = the radius of the vapour molecule,
L = the length of the gradient of gas pressure,
a = v_g/c_g,
ψ_a = a dimensionless function,
α = a coefficient indicating the vapour efficiency.

This equation reveals a good deal by inspection. The ratio p_o/p_f is found to be very small in the normal working of the pump, its value being the smaller for the lighter gases.

In formulating theories of the vapour pump, it has been supposed that the gas molecules are entrained by a gas-free vapour stream emitted from the nozzle. Gaede concluded that there is no theoretical limit to the vacuum obtainable with such a pump, although this does not follow from his formulae. He suggested that in his design the layers of vapour nearest the gap become saturated with gas and therefore can no longer entrain all the gas arriving.

Molthan[4] proved experimentally that the ultimate vacuum of a vapour pump is limited by the partial pressure of gases contained in the vapour leaving the nozzle. These impurities come from the walls or through a leakage. In an ideal pump there is no gas being evolved and no leakage; therefore, the vapour being gas-free, the pump would give the perfect vacuum, an achievement still held possible, at least theoretically, by some recent writers[5,6].

From the above formula, it follows that the ultimate pressure of the vapour pump has a finite value. Moreover, this value may be very low, as in the case of light gases. However, all measurements show much lower vacua than are expected from the theory, particularly with light gases.

Also, the assertion that the perfect vacuum may be achieved by means of the vapour pump is contradicted by the impossibility of obtaining very low pressures either with cascading pumps or with multi-stage pumps. It has been found experimentally that the ratio p_o/p_f normally has a value not smaller than 10^{-3}. This result suggested that some secondary phenomena occur in the working of the pump, so that checking of the theory is impossible. These phenomena are the escaping of gas backwards, which has been noticed by earlier workers[7] and the production of gases and vapours by thermal decomposition of the working fluid[8-11].

It would be essential in testing the theory to be able to avoid the occurrence of the backwards escaping gas, particularly when using light gases. It is the purpose of this paper to describe experiments carried out under working conditions whereby the operation of the pump fulfils the theoretical requirements.

EXPERIMENTAL APPARATUS AND PROCEDURE

In order to obtain the best working conditions and at the same time to be able to control them, an experimental model shown in Fig. 1 has been devised. Its characteristic feature is the way in which the gas enters the vapour. This design does not differ in principle from the common vapour pump. The general arrangement corresponds to that of the vapour pump, except that the vapour flows round the

nozzle through which the gas enters. We see that we have here a somewhat idealised method of producing a blast of vapour, which opposes a tight wall to the gas trying to escape from the fore pressure side*. Any gas escaping backwards passes through the vapour molecules but not along the walls of the pump.

The tube, through which the vapour flows from the boiler, was insulated with asbestos, which contained an additional heater, to avoid any temperature gradient between the boiler and the space N where the pumping action takes place.

The nozzle discharging the gas was provided with internal and external cooling spirals A. The temperature of the vapour at a distance of about 5 mm. below the nozzle was measured by means of a thermocouple. Throughout this work a moderate heating power was used to avoid the decomposition of the fluid. When the pump casing was not water-cooled by one of the external cooling spirals, C or C′, the condensed oil reached the boiler while still hot. The tubes connecting with the backing pump and the McLeod gauge however, were permanently cooled.

The container to be exhausted was an ionisation gauge, type Morse and Bowie[12]. By inserting a liquid air trap between the pump and the ion gauge, vapour could be prevented from having access to the latter. In any attempt to measure the pressure produced by a vapour pump, the vapour pressure of the working fluid must be excluded, as the pump does not remove such vapour.

If in the steady state the gas flow along the connecting tube is negligible, the readings at the gauge will give the pressure produced by the pump. Therefore the values indicated by the gauge are, to some extent, functions of the size of the connecting tube and of any permanent source of evolved or absorbed gas.

Any drop in pressure between the gauge and the front N of the nozzle is actually the result of two processes acting in opposite directions. The first one exists when an appreciable amount of gas is evolved from the walls and internal parts of the gauge, as usually occurs at pressures below 10^{-5} mm. Hg. Final readings were taken when there was no further indication of evolution of gas. This was achieved by letting the pump run and baking the gauge for a long time. This drop of pressure can be evaluated from the fundamental formula for molecular flow and is given by

$$\Delta' p = 1.8 \times 10^{-22} n \qquad \text{(for air)}$$
$$\text{and } \Delta' p = 7 \times 10^{-22} n \qquad \text{(for hydrogen)}$$

where n is the number of molecules evolved by the gauge per second and the dimensions of the connecting tube are taken into account. It is therefore possible to decrease the drop below the lowest pressure measured after a thorough degassing of the gauge bulb, to make n smaller than 10^{15}.

The second process is due to the adsorption of gas molecules by the gauge, and the resulting drop in pressure may be evaluated from :

$$\Delta p = vzp$$

where z is the impedance of the tube connected to the gauge, v is the volume of gas in cm.^{3} at pressure p adsorbed in the gauge per second.

In the above it will be

$$\frac{\Delta p}{p} = 5 \times 10^{-3} v$$

$$\frac{\Delta p}{p} = 1.3 \times 10^{-3} v$$

for air and hydrogen respectively.

The amount of gas cleaned-up in the ionisation gauge is generally much smaller than the value of v which makes the $\Delta p/p$ not more than a few tenths. At the lowest pressure measured, the ratio $\Delta p/p$ approaches unity only if the magnitude of the clean-up phenomenon is equal to a single layer of molecules.

Considering the pumping action of the gauge, the described arrangement is similar to two vacuum pumps acting in opposition. It is obvious that in this case the lowest pressure in front of the nozzle would differ from that indicated by the gauge due to the fall of pressure along the connecting tube (which was no higher than 10^{-7} mm. Hg). That the pump was not

* The suggestion to employ such an arrangement in a practical pump was made, but not published, some years ago by W. J. Scott of British Thomson-Huston Co. Ltd. (Private communication.)

Vol. 4 1954

only working at the lowest pressure measured, but prevailed over any clean-up effect, was indicated by the fact that the indications of the gauge were sensitive to the heating power and the fore pressure.

In our experiments we may therefore assume that the net drop in pressure between gauge and pump was negligible.

EXPERIMENTAL RESULTS

With the device described above, representing a single-stage pump, where the gas entering the vapour does not slide along the walls, it was possible to obtain as low a pressure as is reached usually by means of two- or three-stage pumps. This is a result of great value, as it proves that in the common vapour pump the escaping of gas backwards along the pump casing is unavoidable and can be decreased only by using several stages.

The results obtained give an obvious indication of the existence of gas escaping backwards along the vapour jet, even behind the nozzle. We may assume that this escape gas is present along the whole length of the vapour stream, particularly in the case of light gases.

The curves on Fig. 2 represent the variations of the ultimate vacuum against fore pressure for air and hydrogen, with two different heating powers. The nature of these curves is explained as follows : Below the critical value of the fore pressure, *i.e.* the break-down value, the ultimate pressure is limited by two factors : The rate of the backward escape of gas along the vapour jet and the amount of gas contained in the vapour issuing from the jet.

When the rate of gas escaping backwards is high, as is the case with hydrogen for a low temperature of the vapour, the ultimate pressure increases with the fore pressure (Fig. 2, curve 4). For a higher vapour temperature this increase is noticeable at higher fore pressures only (Fig. 2, curve 2).

If, on the contrary, the amount of gas escaping backwards is negligible, the ultimate pressure is limited by the presence of gas in the driving vapour and therefore is independent of the fore pressure (Fig. 2, curve 1, for air). By decreasing the heating power it is possible to increase the rate of gas escaping backwards until it surpasses the value of the gas impurity in the vapour (Fig. 2, curve 3).

From these experimental results we may conclude that, while operating with air, the rate of the gas escaping backwards can be greatly reduced in the whole range of the working fore pressures, if the heat supplied is sufficiently high. In the case of hydrogen the gas escaping backwards becomes negligible only at low fore pressures and at a higher temperature of the vapour. It is not surprising, therefore, that few commercial pumps make it possible to obtain a very

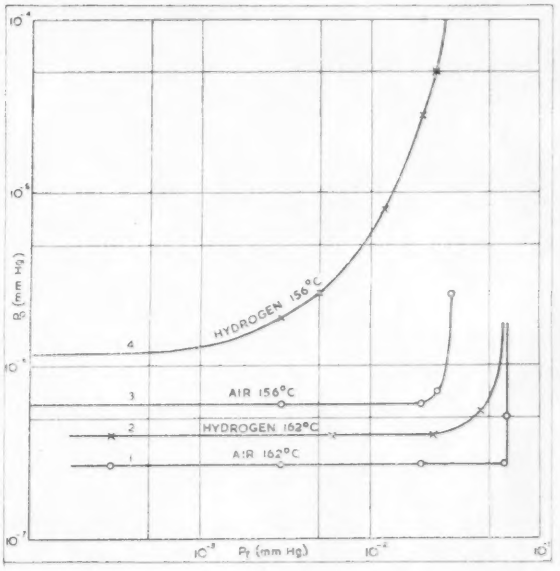

Fig. 2. Variation of the ultimate vacuum with fore pressure, for two different heating powers.

low pressure when working with hydrogen, as long as the normal values of heating power and fore pressure are not increased and decreased respectively.

In practice, the criterion for a well designed pump is that the high vacuum should be independent of fore pressure, when the latter is below a specific value[12], thus preventing the occurrence of gas escaping backwards.

The occurrence of the gas escaping backwards and

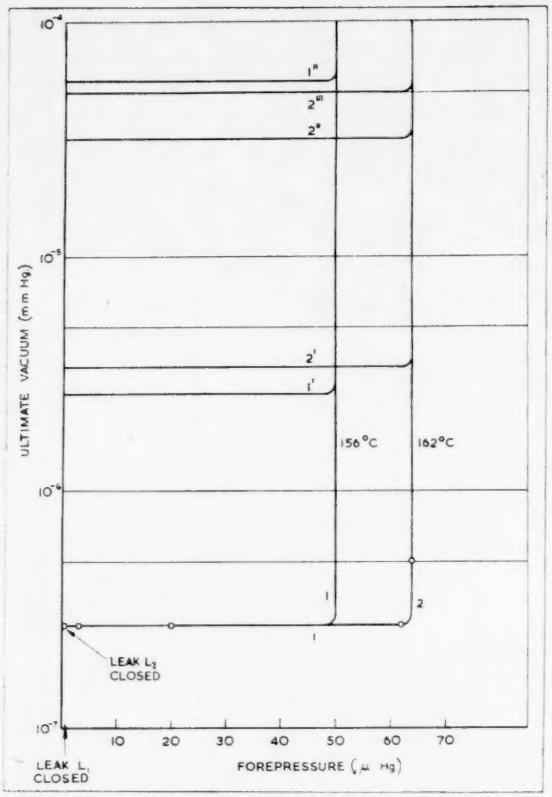

Fig. 3. An illustration of the fact that the ultimate vacuum is limited by the presence of permanent gases in the vapour.

its dependence on heating power and type of gas could be demonstrated by varying the length of the vapour stream. The rubber tube conveying the cooling water could be moved in such a manner that its distance from the nozzle was as short as 1 cm. (position C′) and as far as 12 cm. (position C) respectively. The measured values of the lowest pressure are shown in Table I.

The behaviour of air and hydrogen with respect to the variation of the vapour temperature and the length of the vapour jet shows quite clearly. Thus, the backwards escape is responsible for the gas present in front of the nozzle. The writer suggested before, that this gas escaping backwards does not stop at the nozzle; it travels further along the vapour tube and even reaches the boiler; therefore the vapour stream is never free of gas, particularly in the case of lighter gases. If this be true, then the ultimate vacuum of a vapour pump depends on the design and is a function of the distance between the nozzle and the boiler. For heavy gases and vapours the effect of the gas escaping backwards is practically negligible, so that the ultimate pressure would be affected only by leakage and evolution of gases.

Taking away the cooling coil, the length of the vapour jet was increased only slightly. However, the oil condensed at the bottom of the casing remained hot when returned to the boiler and therefore the amount of dissolved gases in the oil was considerably reduced. In this case the ultimate pressures were found to be lower, both for air and hydrogen (see Table I).

In the case that the amount of gas escaping backwards is negligible, the ratio of the ultimate pressure to fore pressure is constant, up to the critical value. This can be easily proved to be due to the gas contained in the vapour. In Fig. 3, curves 1 and 2 show the lowest pressures which have been obtained with the design described for two different vapour temperatures. If now a small amount of air is admitted through the

Table I

	Lowest Pressure Measured, mm. Hg.			
	Air		*Hydrogen*	
	Vapour Temperature at N		*Vapour Temperature at N*	
	156°C	162°C	156°C	162°C
Water cooling at C′	5×10^{-7}	3×10^{-7}	1.8×10^{-6}	7×10^{-7}
Water cooling at C	3×10^{-7}	3×10^{-7}	7×10^{-7}	6×10^{-7}
No cooling	—	2.7×10^{-7}	—	4×10^{-7}

leak L_2 into the vapour stream, the gauge indicates a higher pressure (curves 1′, 2′, 1″, 2″, 2‴) which remains constant throughout the whole range of fore pressures up to the critical value. Thus, the fact that the shape of curves 2′, 2″, 2‴ obtained after leakage of gas admitted into the vapour stream was similar to that of curve 2 obtained with the leak L_2 closed, suggests that in the latter case also a small amount of gas diffuses continuously into the vapour. This gas is evolved from the walls and from the oil, which dissolves gases all the time. (For similar curves see Ref. 14.)

Measurements of the ultimate vacua, using gases of various molecular weight, would provide valuable conclusions about the working mechanism of the vapour pump. If the ultimate vacuum represents the equilibrium between the rate of the gas molecules transported by the vapour stream and the rate of the molecules diffusing back from the fore pressure side (as the diffusion theory asserts) the lowest pressure obtainable with hydrogen should not be smaller than:

$$p_{o\ \mathrm{hydr}} = p_f \left(\frac{p_{o\ \mathrm{air}}}{p_f} \right)^{0.25} \qquad (6)$$

where $p_{o\ \mathrm{air}}$ is the ultimate pressure for air in the same working conditions as for hydrogen, and supposing that the working fluid is Apiezon B. (See Table IV in a paper previously published by the author[1].)

With the experimental pump previously described measurements of ultimate vacua have been carried out for hydrogen and air. The results in Table II show the huge discrepancy between the theoretical and the experimental values of the ultimate vacuum for hydrogen, which cannot be explained without

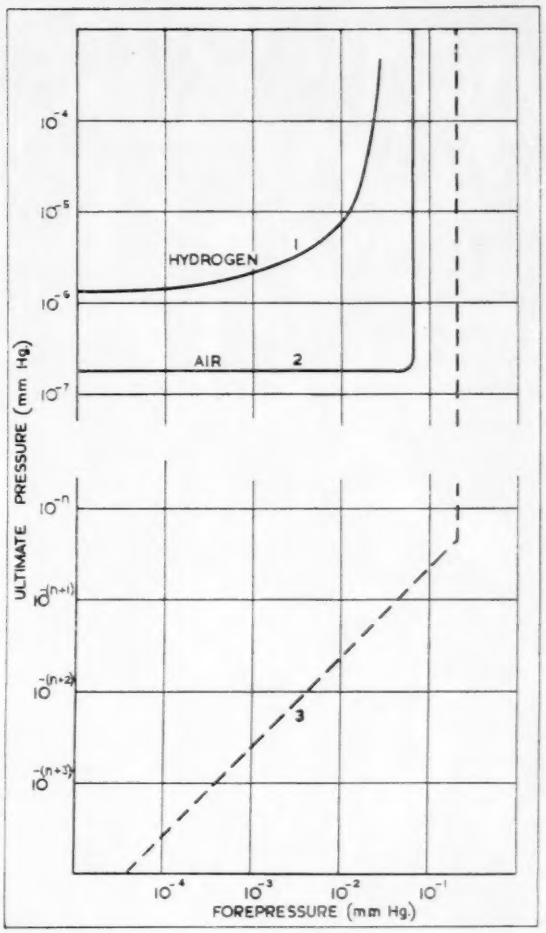

Fig. 4. Variation of the ultimate vaccum with fore pressure for three typical cases: Curve 1: The backward escape of gas prevails. Curve 2: Desorption of gas prevails. Curve 3: The ideal case, n depends on the gas.

Table II

Vapour Temperature °C	P_f *mm. Hg*	$P_{o\ \mathrm{air}}$ *Measured mm. Hg*	*Ultimate Pressure with Hydrogen, mm. Hg*	
			Measured	*Calculated from Equ.* (6)
156	10^{-3}	6×10^{-7}	1.3×10^{-6}	1.56×10^{-4}
156	10^{-2}	6×10^{-7}	6×10^{-6}	9.8×10^{-4}
162	2×10^{-2}	2.7×10^{-7}	4×10^{-7}	1.2×10^{-3}

challenging the validity of the diffusion theory. Therefore, we may assume that the gas returning from the fore pressure side through the vapour streaming in the opposite direction does not obey the law of gas diffusion. This is the effect we call ' backwards escape ' of gas.

The ultimate pressure for hydrogen was lower than the value predicted by the diffusion theory even in the case that the rate of gas escaping backwards was large, a condition, where all clean-up effects are totally negligible.

The fact that the experimental measurements indicate a higher ultimate pressure for hydrogen than for air does not contradict the conclusions of the theory developed by the author, that the ultimate pressure should be lower for lighter gases than for heavier gases. Some investigations have shown that during the running of the oil-vapour pump the decomposition of the working fluid continuously produces permanent gases. Analyses of the residual gases by means of the mass spectrometer[15] at the ultimate vacuum show that in an oil vapour pump, water vapour, hydrogen and hydrocarbons, the latter produced by the pump fluid, are present at the appreciable pressure of about 10^{-7} mm. Hg. The partial pressure of hydrogen and hydrocarbons measured after several weeks' operation and backing for at least 48 hours, reached a limit of about 10^{-8} mm. Hg, although hydrogen is practically absent in the atmosphere, while the air pressure could be reduced to 10^{-9} mm. Hg.

It is obvious that the unavoidable presence of a small amount of hydrogen in oil-vapour pumps at a partial pressure of about 10^{-7} to 10^{-9} mm. Hg makes an accurate measurement of the ultimate pressure impossible. The only way to study the behaviour of a light gas with regard to the lowest pressure obtainable in a vapour pump, would be to use helium in a mercury-vapour pump. This has already been done. Dayton[16] measured the ultimate pressure obtained in mercury pumps for air and helium and arrived at the identical value of 10^{-7} mm. Hg. Thus, although in both cases the diffusion coefficient of helium in mercury vapour is larger than that of air, the ultimate vacuum obtained with helium was not lower indicating that the pressure limit is governed by the presence of gas in the vapour.

CONCLUSIONS

In an endeavour to throw more light on the factors limiting the ultimate pressure of a vapour pump and on the working principle of the pump as a whole, the writer undertook measurements on a pump of experimental design where the gas diffuses into the middle of the vapour stream, so that no cooling is required for the pumping action. Therefore the phenomenon of vapour condensation does not play any part in the working of the pump. In fact, cooling just below the point where the gas enters the vapour stream greatly reduces the performance of the pump as far as lighter gases are concerned (see Table I).

Comparative measurements of the lowest pressures for air and hydrogen demonstrate that the dependence of the ultimate vacua on the fore pressure does not fulfil the theoretical requirements of the diffusion theory. Even when the amount of gas escaping backwards was appreciable, the lowest pressure measured when pumping hydrogen was much lower than calculated.

The present investigation shows that two secondary processes occur all the time in a vapour pump. It was found that both, the backward escape of gas and evolution of gas (mainly from the oil) are responsible for limiting the ultimate vacuum obtained.

Pumping hydrogen and air respectively and controlling the vapour temperature (see curves 1 and 2 in Fig. 4) it was possible to establish working conditions, in which either phenomena prevailed. Which one of the two was predominant could be determined from the shape of the curves showing variation of the lowest pressure against fore pressure.

We may assume that in a well designed vapour pump the ultimate vacuum is limited, not by the pressure of the gas diffused from the fore pressure side which is a condition of the diffusion theory and accepted even in a very recent paper[17], but by the lowest total pressure of all gases and vapours already contained in the vapour before it leaves the nozzle, apart from the partial pressure of the vapour of the

Vol
4
195

working fluid.

For an ideal pump, in which no secondary phenomenon occurs, the variation of the ultimate vacuum is indicated by the curve 3, in Fig. 4, as derived from Equ. (5).

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THE ULTIMATE VACUA OF TWO-STAGE ROTARY OIL PUMPS

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Summary

IT HAD BEEN NOTICED THAT rotary pumps show an increased ultimate pressure as their temperatures rise during operation. It is established that this pressure rise is not due to the presence of vapours of the oil used in the pump itself but possibly to the progressive appearance of vapours from the lighter fractions of the oil liberated as a result of decomposition. It is concluded that this phenomenon resulted from the thermal effects of the vanes sliding in the rotor where the condition of boundary lubrication exists. Extreme pressure additives to the oil had a marked influence on these pressure variations and it may be possible to discover an additive which will completely prevent the cracking of the oil.

Sommaire

ON S'EST APERÇU que les pompes à palettes présentent une diminution de vide maximum, pendant que leur température augmente en cours d' utilisation. Il a été établi que cette augmentation de pression n'est pas dûe à la présençe de vapeurs de l'huile utilisée dans la pompe, mais peut être bien à l'apparition progressive de vapeurs provenant des plus légères parties de l'huile, libérées par décomposition. On en conclut que ce phénomène est dûe à l'effet thermique engendré par les palettes, coulissant dans le rotor, où le phénomène de lubrification limitée existe. L'addition à l'huile de produits pour la lubrification de surfaces coulissant sous de très grandes pressions mécaniques, à un effet certain sur les variations du vide dans la pompe, et il est peut être possible de découvrir un produit qui empêchera complètement la décomposition de l'huile.

INTRODUCTION

THE FOLLOWING SETS out the results of experiments aimed at determining the performance of oils in two-stage rotary vacuum pumps. Very little information is available about the composition and structure of these oils. It is generally understood that in the main the oils are natural mineral oils selected and blended to give a viscosity which pump manufacturers consider satisfactory and also to have a low water and sulphur content. The mean molecular weight of the oils is said to be about 400. Having this high molecular weight, the fact that they are oils and not waxes indicates that the molecules are not straight aliphatic chains. It is also thought that there are few double bonds in the molecular structure, *i.e.*, they are largely saturated hydrocarbons.

The pressure developed by these pumps is well known to consist of two parts. One is that of permanent gases and is measurable on a McLeod gauge. This pressure never exceeds 10^{-4} mm. Hg in a well assembled two-stage pump, and may be much lower. The second part is that which is measurable on Pirani or Alphatron gauges and is called generally 'oil vapour'. This pressure may be anything from 10^{-3} mm. Hg upwards to about 10^{-2} mm. Hg, depending on conditions. It is best measured with the Alphatron since it is insensitive to an amount of contamination sufficient to cause a Pirani gauge to drift seriously.

No mention of the nature of this vapour has been found in the literature.

† MS. received August, 1955.

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The experiments to be described were carried out at first with a pump having an Alphatron connected by about a foot of glass tubing. As the experiments proceeded it became apparent that the temperature of the pump varies with time after starting up and that this was accompanied in some pumps by an increase in pressure reading. As a result of this it was decided to heat the pumps artificially by placing heaters under them to determine the relation between pressure and temperature and it was then found that in all cases the pressure increased with increasing temperature.

This result is remarkable in that the temperature of the gauge and the tube connecting it to the pump remained at constant temperature, indicating that the pressure reading is not in fact that of a condensable vapour in equilibrium. It is probably due to the products of disintegration of the oil molecules owing to the action of the pump. If one takes into consideration the volume of the connecting tube and gauge one will realise that a few micrograms of the pump oil disintegrating into ethylene, acetylene or similar fragments could set up the pressures observed.

An attempt to measure the vapour pressure of the oil with a thermionic ion gauge in a sealed-off system gave readings well below 10^{-4} mm. Hg at first, but as the gauge decomposed the oil, the pressures rose. It is concluded therefore that the true oil vapour pressure is very low and can only be measured properly with a Knudsen gauge, which might be made sufficiently sensitive without the heaters reaching a temperature which would cause appreciable decomposition of the oil molecules.

The measurements recorded must be viewed only as readings on the Alphatron. Since the nature of the 'vapours' or decomposition products is not known and all ionisation gauges including the Alphatron are sensitive to the molecular weight and structure of the ionised medium, the readings are certainly not true pressure readings.

PERFORMANCE OF ROTARY OIL PUMPS

Variations in Ultimate Pressure

All experiments were carried out on pumps which had been assembled and checked so as to give residual pressures of less than 10^{-4} mm. Hg on a McLeod gauge.

The experiments were carried out in the main on several examples of two types of pump—the Edwards Speedivac 2B and the Pulsometer 2D1. All these pumps had seen many years of service and were cleaned and reassembled many times in the course of the experiments. Occasionally they were cleaned with alumina powder to ensure as far as possible for some experiments that no trace of other oils were left. The ultimate Alphatron reading obtained was usually about 4×10^{-3} mm. Hg. Successive runs on a particular pump and oil often gave considerable differences in ultimate vacua (see Figs. 2 and 10, for example). It came to be regarded as necessary however that a good pump and oil would invariably give less than 10^{-2} mm. Hg. Sometimes readings down to 1×10^{-3} mm. Hg were obtained. It was fortunate that the readings came just within the range covered by the Alphatron.

Stepped Vapour Pressure Curves

Curves of pressure against time for a Pulsometer 2D1 exhibited a sudden rise or step after running for about an hour (see Fig. 1). At the end of the run the temperature of the pump was of the order of 40°C. This rise was thought to be due to the increase of running temperature and it was found that when the pump was heated above the maximum running temperature, a whole series of these steps occurred. The positions of the steps along the time axis were very nearly repeatable (see Fig. 2), although the pressures at which they occurred varied, depending on the lowest ultimate vacuum obtained during the run (see previous section). It was also noticed that the sizes of particular steps were fairly consistent during a series of runs.

Speedivac 2B pumps were found to exhibit these steps at a higher temperature than the Pulsometer 2D1 (Fig. 3).

No satisfactory explanation could be found for either the variation in ultimate vacuum, or the occurrence of the stepped temperature/pressure curve. The difference between Speedivac and

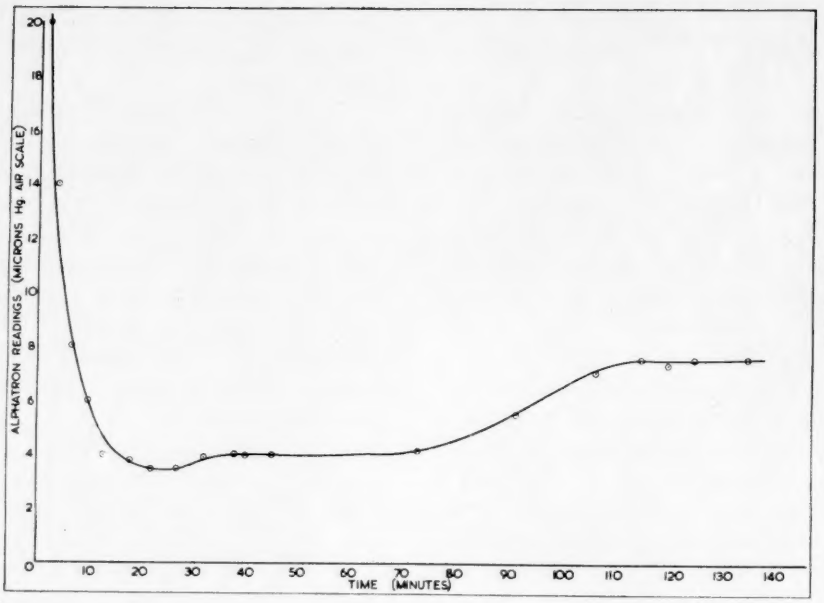


Fig. 1. Pressure/time curve of a Pulsometer pump, Model 2D1, operated with Dicks No. 2 oil, exhibiting a pressure rise after about 1 hour operation.

Vol. 4 1954

Pulsometer pumps was particularly curious and suggested that the mechanical design of the pump was responsible. A detailed comparison of the design of these pumps was made, the most significant difference being that the blades of the Pulsometer were of the same hardness as the chamber, whilst the blades of the Speedivac were very much harder than the chamber.

Various experiments such as removing the 'hat leather' sealing off the shaft between the two chambers in the Pulsometer pump, an analysis of the materials of which the pump was made and a study of the mechanical finish gave no clear indication of the reason for the difference between these pumps. It is to be noticed that the more recent design of Speedivac pump, the 2S50 Model, gives a performance similar to that of the Pulsometer pump, even though the materials and finish of the earlier and the later Speedivac pumps are superficially similar.

MODERN THEORIES OF LUBRICATION

A detailed account of the theories of lubrication is given by Bowden and Tabor[1]. It is seen that the main types of lubrication are :

(*a*) *Hydrodynamic*

Here a continuous oil film separates the sliding surfaces and there is no metallic contact between them.

(*b*) *Boundary*

In this case the lubricating film is so thin that it is of molecular dimensions and is so closely in contact with the surfaces that it may be considered to be no longer fluid. There is a certain amount of 'break-through' of the lubricating film by surface asperities, so that as the surfaces slide upon each other, metallic wear occurs and high temperatures are developed at the asperities. These temperatures may be as high as the melting point of the sliding material. Special oils containing additives of various types tend to reduce the friction between the surfaces in spite of the extreme pressure.

Lubrication between Pump Chamber and Rotor Blades

If we assume that boundary lubrication exists, then it seems likely that the extreme pressure and heat

developed during welding and shearing the surface asperities will cause cracking of the lubricating molecules. This will cause the liberation of hydrocarbons of lower molecular weight, which could possibly be gases such as the lowest members of the unsaturated aliphatic series. The liquid oil will be enriched with components of lower molecular weight and also become slightly unsaturated. Thus, not only will the oil acquire components with a higher vapour pressure, but more or less permanent gases will be liberated in the pump.

It is tempting to try to explain the steps in the temperature/pressure curves by means of a hypothesis based on the progressive appearance of vapours due to liquid fractions of very low molecular weight. In view of the random character of the action taking place and the complicated nature of the oil however, it appeared unlikely that such an explanation could be substantiated in detail. The fractions yielding the steps in vapour pressure are presumably those of the lighter liquid products formed by breaking down the heavy oil molecules and are not those of the heavy oil fractions themselves. The oil vapours condense in the Alphatron, to the extent that the radioactive source occasionally became contaminated and the gauge required cleaning. In spite of this however, for many runs after such cleaning the gauge would repeat low readings such as those quoted on page 41. The pressure steps observed, then, appear to be due to various fractions of the very light components being successively evaporated due to friction and going over into the Alphatron and dissolving in oils already present. The pressure observed is not that of saturated vapours but of an unsaturated vapour flowing from the pump into the gauge, which was always cooler than the pump.

Early in the investigations an oil having a high sulphur content was found to give a consistently lower vacuum than ordinary oils. Since the high sulphur content was outside the specification, the oil was rejected as unsuitable. Further consideration in the light of the ideas on lubrication expressed above suggested that the sulphur was acting as an extreme pressure (E.P.) additive and we were encouraged to investigate the effects of other additives on the performance of rotary oil pumps.

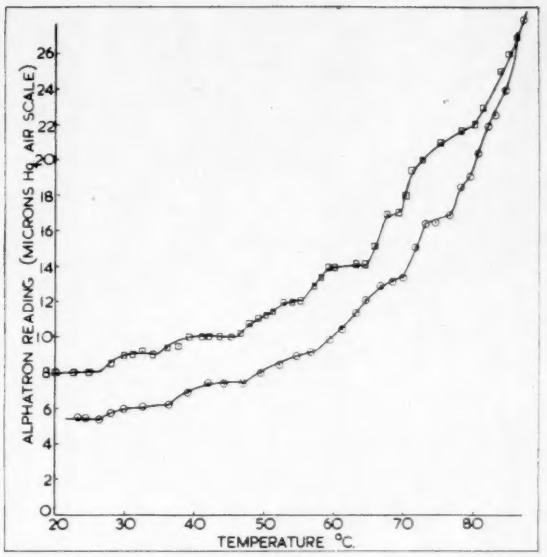
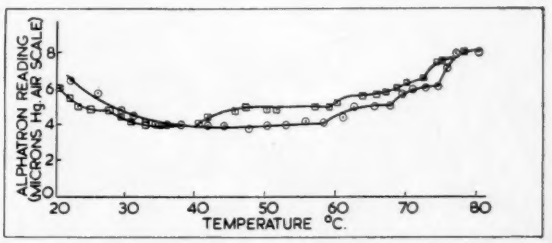


Fig. 2. Pressure/temperature curves of 2 Pulsometer pumps, Model 2D1, operated on Edwards No. 8a oil, indicating a series of pressure rises on heating above maximum running temperatures.

Experimental Evidence of Boundary Lubrication

It was decided to examine the mode of lubrication existing in the pump chamber by a method similar to that used by Courtney-Pratt[2] for the examination of cylinder lubrication of an internal combustion engine. Thin films of oil exhibit high electrical resistance, whereas when metallic contact exists the resistance is, of course, low. By applying a voltage across the

Fig. 3. Pressure/temperature curves of 2 Speedivac pumps, Model 2, operated on Edwards No. 8a oil indicating the same phenomena as demonstrated in Fig. 2 but at higher temperatures.



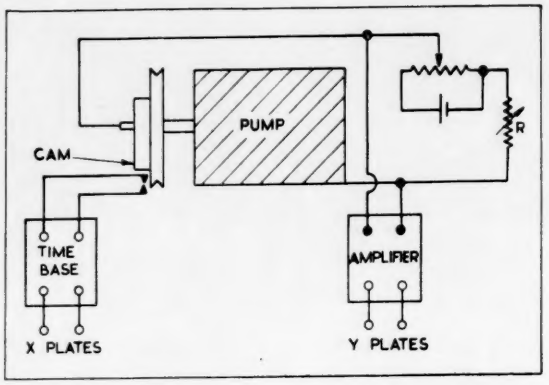
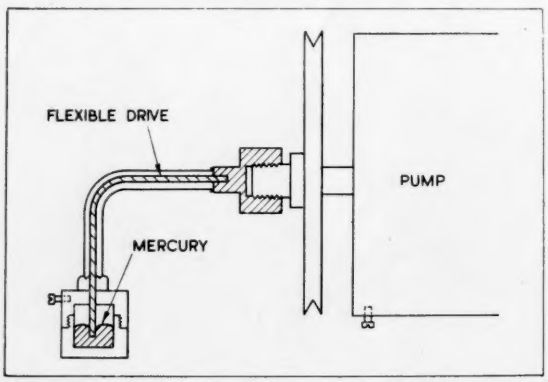


Fig. 4. Diagram of the experimental arrangement employed to determine the mode of lubrication prevailing in the pump chamber. *Note :* For calibrating, R is at least 10× residual circuit resistance.

moving parts and examining the conduction of electricity it is possible to see where a 'breakthrough' occurs. A time base representing one complete revolution of the pump was used to obtain the horizontal deflection of an oscilloscope, the vertical deflection being obtained from the voltage fluctuations across the rotor and stator *via* a suitable amplifier. A resistance R was included for calibration purposes (Fig. 4). Contact to the rotor was made *via* a flexible lead terminating in a mercury pool (Fig. 5). A camera was used to record the oscilloscope trace.

Using this apparatus, hydrodynamic lubrication is represented by maximum vertical deflection, and complete breakdown is represented by zero deflection.

Fig. 5. Detail of arrangement shown in Fig. 4, showing method of providing electrical contact to pump rotor.



By this method it was possible to establish that the journal bearings, being lightly loaded, were subject only to hydrodynamic lubrication and that the pump chamber was subject to boundary lubrication. Figs. 6, 7 and 8 show typical lubrication conditions occurring in a rotary oil pump using a straight mineral oil. From such curves however it was not possible to predict with certainty any of the steps in the pressure/temperature curve.

These photographs are the result of single shot exposures, but except for the fine details of the effects due to breakdown of the film, each revolution of the pump produces almost identical oscillograms.

EFFECTS OF EXTREME PRESSURE LUBRICANTS

The following E.P. additives were thought by the Regent Oil Company to have a sufficiently low vapour pressure to be of use in vacuum pumps and were mixed by them with a straight mineral oil:—

(*a*) Lead Naphthenate
(*b*) Chlorinated Wax
(*c*) Straight Antoxydant

Lead Naphthenate

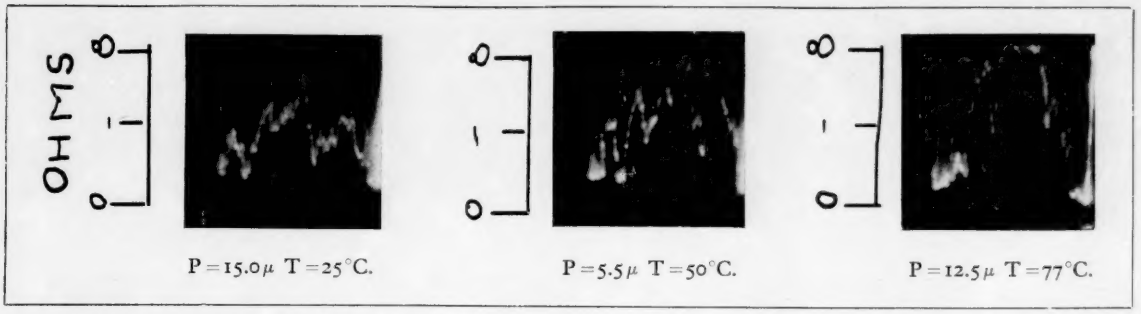
This additive was found to yield an oil having a very high vapour pressure. Since it was useless as a pump oil, it was not examined in detail, although the lubrication of the chamber appeared to be slightly improved. Fig. 9 shows a pressure/temperature curve for this oil.

Chlorinated Wax

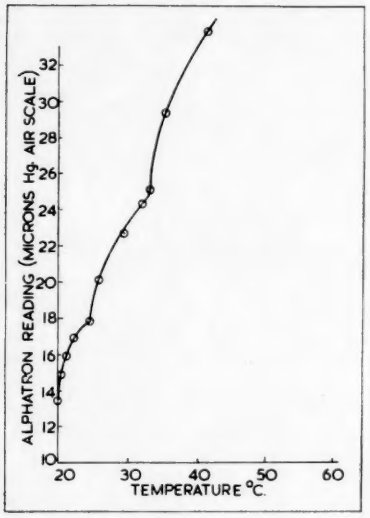
Fig. 10 shows the pressure/temperature curve for this additive. It is seen to be rather different from the previous graphs. The lowest pressure obtained, 1.5×10^{-3} mm. Hg, is very good. The steps have disappeared and have been replaced by a series of fluctuations. Figs. 11, 12 and 13 show that the lubrication in the pump chamber has been considerably improved compared with Figs. 6, 7 and 8.

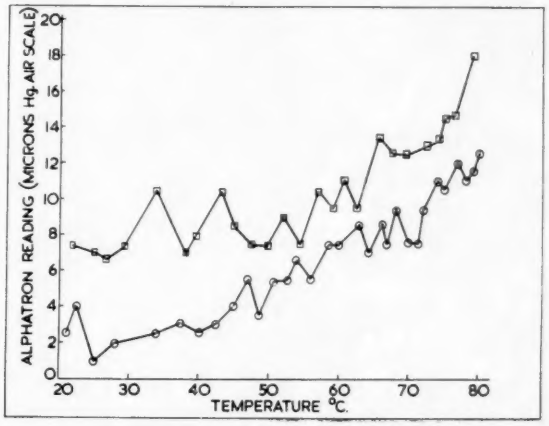
Straight Antoxydant

Fig. 14 shows an unexpected characteristic. The



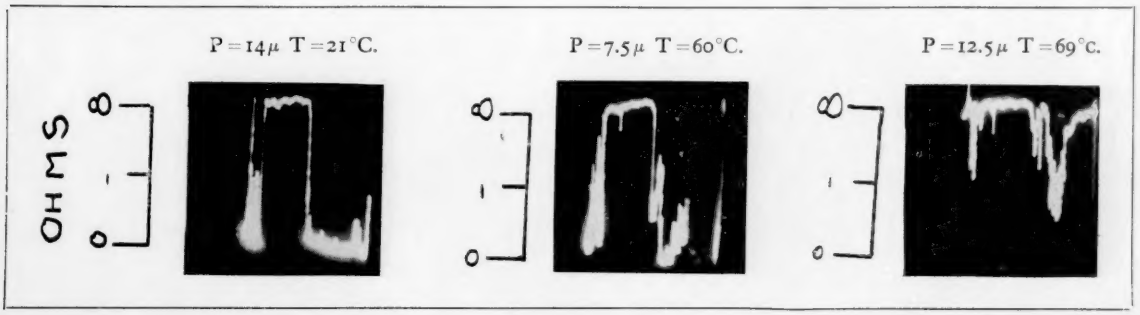
Figs. 6 7 & 8 (*above*). Oscillographs recording lubrication conditions, typical for rotary pumps operated on a straight mineral oil (Regent 7030).





Pressure/temperature curves of a Pulsometer 2D1 pump operated on oils with various additives:— *Fig.* 9 (left): Regent 7030 + Lead Napthenate. *Fig.* 10 (above): Regent 7030 + Chlorinated Wax (2 runs on the same pump).

Figs. 11, 12, 13 (below). Oscillographs recording lubrication conditions, obtaining during runs with Regent 7030 + Chlorinated Wax (see Fig. 10).



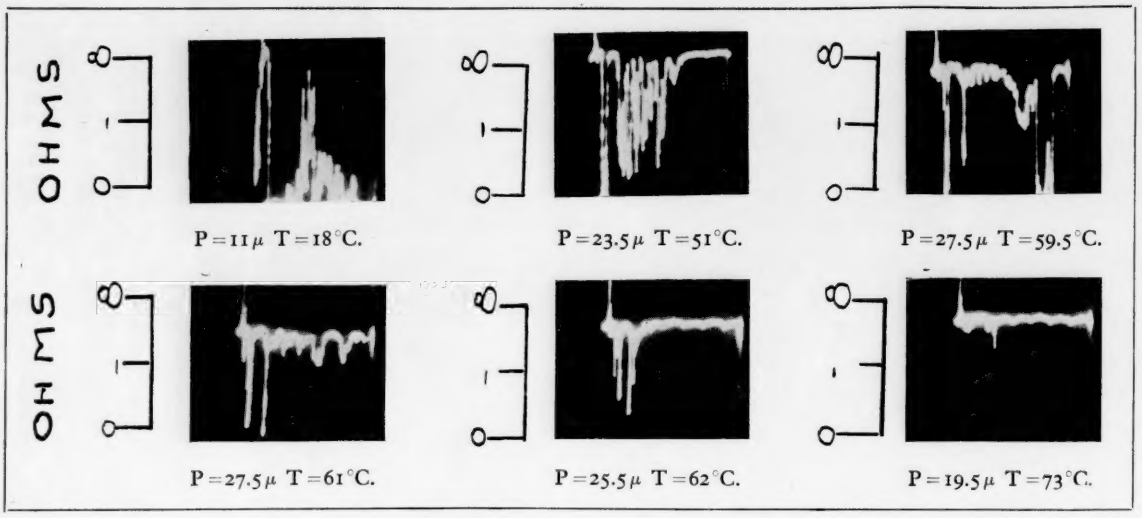
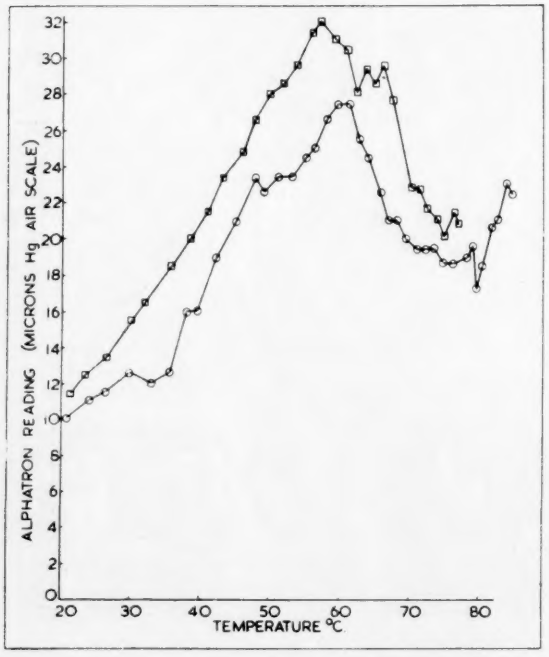


Fig. 14 (below). Temperature/pressure curves of a Pulsometer 2D1 pump operated on Regent 7030 + Antoxydant.

Figs. 15, 16, 17, 18, 19, 20 (above). Oscillographs recording lubrication conditions prevailing during runs on Regent 7030 + Antoxydant (see Fig. 14). The lubrication is shown to change from boundary to hydrodynamic.



lowest pressure was 10×10^{-3} mm. Hg which although not good is not unduly high (see page 41). The pressure rises in the usual manner until a maximum of 28×10^{-3} mm. Hg is reached at 60°C; it then falls (with fluctuations similar to those of the chlorinated wax) to 18×10^{-3} mm. Hg at 80°C, after which it rises again. The most interesting fact here is that the character of lubrication changes from boundary to hydrodynamic when the maximum is reached. (See Figs. 15–20.)

CONCLUSIONS

All the evidence collected indicates that a considerable part of the pressure obtained from a rotary oil pump is due to gaseous and liquid products of decomposition of the oil molecules brought about by the very intense local heating of the pump surfaces resulting from friction and high (mechanical) contact pressure.

The marked difference between the Speedivac 2B on the one hand and the Pulsometer 2D1 and later Speedivac models is not explained.

The stepped characteristic of the pressure reading *versus* temperature curve is possibly due to the progressive appearance, at measurable pressures, of vapours originating from the lighter fractions of the

Vol
4
195

oil resulting from decomposition. The pressures observed are in any case not the saturated vapour pressures of these fractions.

The effect of chlorinated wax as an E.P. additive is that the steps are removed and replaced by fluctuations in the temperature/pressure curve; associated with this is a distinct improvement in the number of ' break-throughs '. In the case of the antoxydant the sudden drop in pressure is accompanied by an abrupt change of lubrication mechanism.

Fluctuations in pressure seem to be a property of the additives tested and suggest that they are not entirely satisfactory, but the experiments give hope that an additive may be found which will appreciably reduce the cracking of the oil. In such an event it could be expected that the vapour pressure of the oil would be the true limit of vacuum for these pumps.

The Authors are indebted to Mr. R. G. Gilson and Mr. A. D. Cassell for the valuable assistance given during the experiments. Thanks are due to Mr. N. G. Gullick and the Regent Oil Company for their ready co-operation, and to Mr. B. Wilkinson, Managing Director of Electronic Tubes Limited, for permission to publish this paper.

Vol. 4 1954

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the current situation of FREEZE DRYING OF SEMEN

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Summary

EXPERIMENTS of freeze drying bull semen are reported. On reconstitution motility was observed but the fertilising capacity was not investigated. The effects of glycerol, used as the protective agent, are discussed. Some freeze-dried spermatozoa show a morphological change rarely observed in other methods.

Sommaire

DES EXPERIENCES sur la lyophilisation du sperme de taureau sont rapportées. A la reconstitution des particules mobiles furent observées, mais le pouvoir fertilisant ne fut pas étudié. Les effets du glycérol, employé comme agent de protection, sont discutés. Quelques spermatozoides lyophilisés montre un changement morphologique rarement observé dans d'autres méthodes.

Vol. 4 1954

INTRODUCTION

THE GREAT PROGRESS of the freeze drying technique, its successful application in the case of living cells and the increasing use of the artificial insemination method have made a study of the freeze drying of semen worthwhile. For some time a method has been sought which would facilitate the preservation of semen without reduction of its fertilising capacity. The discovery a few years ago, of the protection afforded by glycerol when freezing certain types of cells made it possible to preserve semen for protracted periods at a temperature of -79°C but this method is still very new and as yet it is not known how long semen may be preserved in this way without losing its fertilising capacity. While it appears that this method presents a solution of the problem of preserving semen to be used in artificial insemination for a long period, there are some difficulties in the practical use of that method. The method is expensive and there are technical difficulties in maintaining semen for more than a short period at the low temperature required. In the case of the freeze drying method it has been possible to preserve living cells such as bacteria at ordinary refrigerator or even at room temperature. Thus, if the latter method were successful as far as the preservation of semen is concerned, the difficulties just mentioned could be avoided.

Previous work on the freeze drying of living cells, especially those of metazoa, has shown that the most severe damage to the cells occurs during the freezing process. Sperm cells can be frozen successfully. Thus, the first of the three steps of the freeze drying method, which consists of freezing, drying and reconstituting, can be accomplished satisfactorily, but comparatively little work has been carried out on the reaction of semen to the drying operation. Up to the present, successful experiments have been reported in one case only, *viz.*, fowl semen[1]. In that case motile spermatozoa were found in semen reconstituted after freeze drying. The semen, diluted with Ringer's solution containing 20% glycerol in a proportion of 1 : 1, was frozen at a temperature of -79°C. After allowing the temperature to rise to -25°C the semen was dried employing a condenser filled with liquid

* MS. received January, 1956.

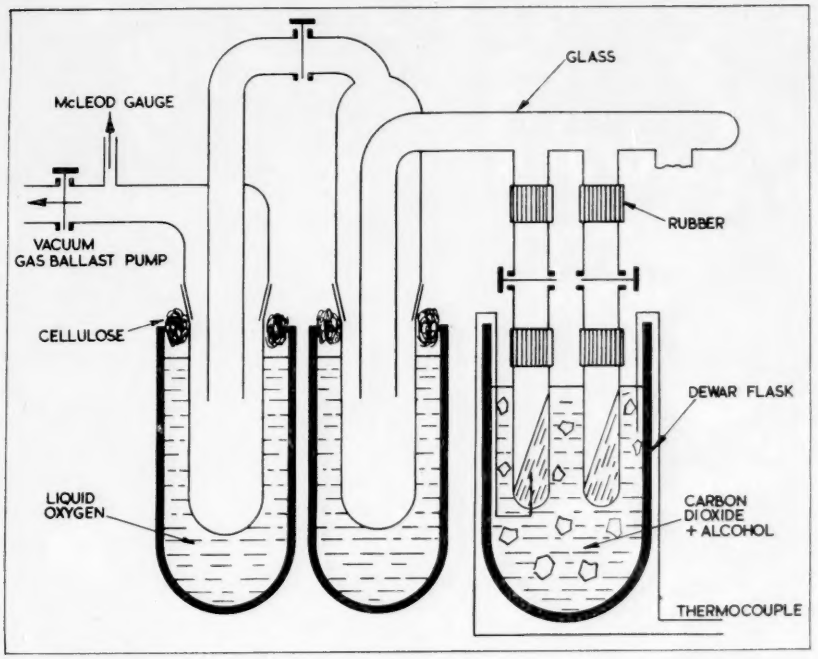


Fig. 1. The freeze drying equipment used in the experiments with bull semen.

air. On reconstitution of the apparently dry material up to 50% of the spermatozoa preserved were found to be motile. Unsuccessful attempts at freeze drying bull semen are reported by Schmit and Neumann[3] and human semen by Sherman[4].

FREEZE DRYING OF BULL SEMEN

Experimental Method

The freeze drying of bull semen has been investigated in this laboratory. The freezing method employed generally followed that described by Polge and Rowson[2]. The semen was diluted at + 20°C with a 3.92% sodium citrate solution containing 30% egg yolk in a proportion of 1 : 3. The diluted semen was cooled down slowly to +5°C over a period of 2–3 hours. Then the diluted semen was mixed with so-called 'glycerol diluter' (3.92% sodium citrate solution with 20% by volume of glycerol and 20% of egg yolk) in a proportion of 1 : 1.

A period of 12 hours was allowed for equilibration of the semen with the glycerol. Subsequently the semen was placed in the drying vessel and frozen at a temperature of –79°C. When this temperature was reached drying was started and the temperature permitted to rise gradually at the same time. The equipment used is shown in Fig. 1. The condenser was operated with liquid oxygen. In these conditions drying took 4–5 days. The liquid oxygen charge had to be replenished usually at intervals of 3–4 hours. When the apparently dry material obtained by this method was reconstituted, motile spermatozoa were found on completion of several, but not of all, experiments. Only relatively few motile spermatozoa could be observed where the experiments were successful and thus it was not possible to carry the investigation any further by testing the fertilising capacity of the reconstituted semen.

Experimental Results and Discussion

These experiments demonstrate that bull spermatozoa can withstand successfully the withdrawal, at low temperatures, of a large amount of the water contained in the semen. This has not been previously

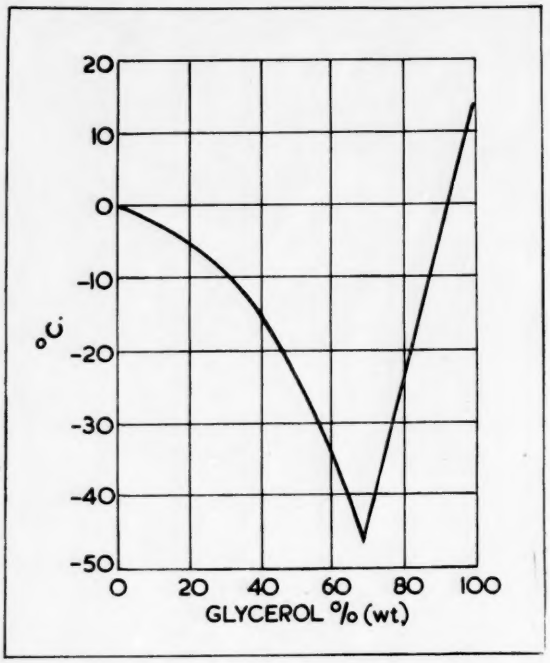


Fig. 2. Freezing points of glycerol-water solutions (from, Harris, R. J. C., *Biological Applications of Freezing and Drying*, Academic Press, Inc., New York, 1954).

demonstrated in the case of the semen of mammals. It is not yet possible to give a precise indication of the degree of water withdrawal which will cause severe damage to the spermatozoa. The dehydration of the semen occurs at varying rates in different parts of the sample. If the process is interrupted in the initial stage, only an average value of the degree of dehydration obtained can be determined. If motile spermatozoa are observed after reconstitution of the initially dried material it can be assumed that they were contained in the portion of the sample which was still frozen. In the second stage of drying however, when the material as a whole is no longer frozen, the sample is more porous and comparatively homogeneous with respect to its moisture content. At this stage, therefore, the water content measurements apply to all parts of the processed material.

At the conclusion of the first drying stage the water content of the material is about 5%. During the second stage the water content decreases still further. The final moisture content was determined by drying the processed material in an oven at 100°C until no further change in weight occurred. In our investigation we found no motile spermatozoa in samples, the drying of which was continued beyond a short period in the second stage. The progress of dehydration was checked by means of two thermocouples, one inserted into the sample and the other into the cooling fluid. As long as water was evaporated in significant quantities the thermometer in the material subjected to drying indicated a lower temperature then the thermometer in the cooling fluid, due to the heat extracted by the evaporation process. But the values obtained are applicable only to the part of the sample surrounding the thermometer. If the thermometer happens to be in the right position the values recorded will serve the purpose, for which they are intended. Without such temperature measurements in the drying vessel no indication is given of how dehydration is progressing and at what point the first drying stage is completed. These measurements are specially important when the work is carried out in laboratories where processing conditions are rarely constant.

As already mentioned the material is kept at a low temperature during the dehydration. The temperature must remain below the freezing point as thawing may result in injury. But the value of the freezing point changes continuously during the dehydration period. In theory, since glycerol is employed as a protective agent, the working temperature should remain all the time below the freezing points of the mixture at the various concentrations occurring while dehydration is in progress, see Fig. 2. This agrees with the experimental data since in our studies living spermatozoa were found, only when dehydration was carried out at temperatures below those shown in Fig. 2.

Due to technical difficulties it was not feasible to carry out the entire process at −79°C. For this reason and in order to shorten the time required for dehydration, experiments were carried out to determine whether the final stage of the drying process could be completed successfully at −25°C.

Vol. 4 1954

The timing of the changeover from the lower to the higher temperature is a critical factor in such experiments. It depends on the percentage of glycerol in the mixture but there is no practical method yet in existence which permits continuous determination of this quantity while dehydration is in progress. This difficulty of effecting the change of temperature at the correct time is the chief cause for the variations in the results obtained.

EFFECTS OF GLYCEROL

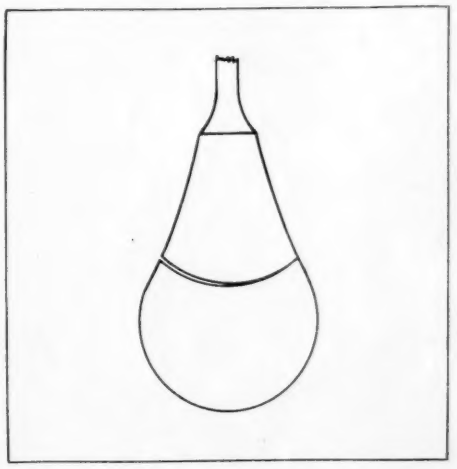
Although glycerol is a powerful means of preventing injury to the spermatozoa during the freezing process, it has two disadvantages as far as dehydration is concerned : Firstly, glycerol-water mixtures have comparatively low freezing points and this prolongs the processing time. Secondly, glycerol appears to be toxic with respect to spermatozoa at concentrations as high as those normally occurring on the conclusion of the dehydration.

Before diluted bull semen is mixed with glycerol-diluter, it contains about 12% water-free solids. After addition of glycerol in the usual 1 : 1 proportion the mixture contains about 16% water-free solids. Disregarding the small amount of salt contained in the glycerol-diluter the water-free solids in the glycerol-semen mixture are made up in the proportion of two parts of glycerol to one part of semen. Therefore, the dried material prepared in this way has a consistency similar to that of cold honey. It is not a powder and its reconstitution is more difficult than that of powder. Theoretically, the disadvantages presented by the use of glycerol may be avoided if one employs another equally effective protective solution or if the glycerol is removed after equilibration. We have tried both ways without success. For example, we used concentrated sugar solutions as an alternative to glycerol but their protective action during freezing was insufficient. Further, we removed glycerol after the equilibration stage by centrifuging the material at 4,000 r.p.m. in a centrifuge refrigerated at +5°C which is the temperature at which the mixture has to be kept after addition of the glycerol. No motile spermatozoa could be traced after freezing. On the other hand we found that this technique of removing the glycerol, in itself, had no harmful effects because samples to which glycerol-diluter was added again after centrifuging, showed the same number of motile spermatozoa after freezing as the controls.

MORPHOLOGY OF FREEZE-DRIED SPERMATOZOA

According to the results of investigations of other workers no morphological changes could be discovered after freeze drying. Superficial observations in the course of our experimental work also failed to reveal any effects of freeze drying on the morphological structure of spermatozoa. On occasions more separated heads than normal were found but this may happen any time due to purely mechanical effects such as those prevailing when a smear preparation is made. A more careful study, however, revealed that almost every sample of semen treated by the freeze drying method contained a certain percentage of spermatozoa showing a peculiar morphological change, *i.e.* a crack around the middle portion of the spermatozoon as indicated in Fig. 3. This phenomenon is only very seldom observed in the spermatozoa processed by other methods. Therefore

Fig. 3. Bull spermatozoon showing a crack near the middle of the body after freeze drying.



freeze drying must have an effect on the spermatozoa which the other methods have not. On the other hand coiling of tails and similar changes which occur frequently in association with oscillations of osmotic pressure were not observed. Since, in this method, dehydration is carried out below the freezing point, changes associated with fluctuations in osmotic pressure would not be expected to occur during the drying period but it is surprising that such changes do not appear during the reconstitution process.

Further work on the effects of certain factors in the freeze drying of semen is in progress.

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Vol. 4 1954

VACUUM STANDARDS

STANDARDS FOR VACUUM FITTINGS

Summary

A REPORT ON A SYSTEM of vacuum flanges sealed by O-rings which is being considered for presentation to the American Standards Association by a committee of the technical society 'The Committee on Vacuum Techniques' for adoption as an American Standard. Comments are solicited by the author.

Sommaire

UN RAPPORT sur un système de brides pour appareil à vide, dont l'étanchéité est assurée par un joint circulaire de caoutchouc, et dont la présentation par un comité technique du 'Comité des Techniques du Vide' à l'American Standard Association est prévue pour être adopté comme standard Américain. L'auteur désire des commentaires.

The author is the chairman of a committee of the recently organised technical society 'The Committee on Vacuum Techniques.' This sub-committee is charged with drawing up Standards for Vacuum Fittings for eventual consideration by the American Standards Association. The decisions which are to be made are of obvious concern to experimental physicists, who are not only users, but also designers and builders of vacuum equipment. It has become apparent in our physics laboratory that a considerable fraction of the time of our machine shop and of our experimental physicists is going into the design and construction of vacuum fittings. Fittings are designed and made for each particular job with no attempt at standardisation. This is obviously a procedure very wasteful of time and money.

If Standards could be agreed upon, it would be possible for manufacturers to make fittings and have them in stock for their own use and for sale to users of vacuum equipment. As a result parts would be interchangeable and different pieces of equipment would always fit. However, for these Standards to be of greatest use it is necessary that many physicists be aware of this work and contribute to it as many practical suggestions as possible, before the Standards are agreed upon.

The committee is first taking up the standardisation of O-ring-sealed flanges. If these can be decided upon, fittings can be designed around them. Flanges for ultra-low-pressure work will be considered later but suggestions for them will be welcomed.

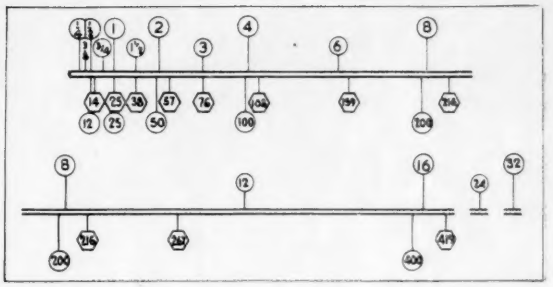


Fig. 1. Comparison of the nominal sizes of fittings. The upper circles give a suggested 'first series' of inch sizes. The second set of circles give a 'secondary series' of inch sizes. Below the line in hexagons are the millimetre sizes under consideration by the French committee. At the bottom is a suggested metric series which joins easily to the 'first series' of inch sizes.

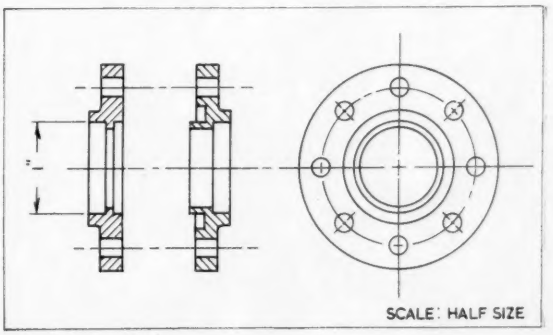
The system of flanges under consideration has been based on the following design principles for fittings :

(1) It must be possible to use all reasonable metals for fabricating fittings and flanges, *i.e.* steel, brass, copper, duraluminium, nickel, Monel and stainless steel.

(2) Welding and brazing shall be used for putting flanges on tubing and fittings. Suitable techniques are now available for welding and brazing all the above metals, so there is no need to struggle to avoid them.

(3) Since the vacuum industry is really a small industry, it is very important to have the

Fig. 2. Scale drawing of 1-inch bolted flange.



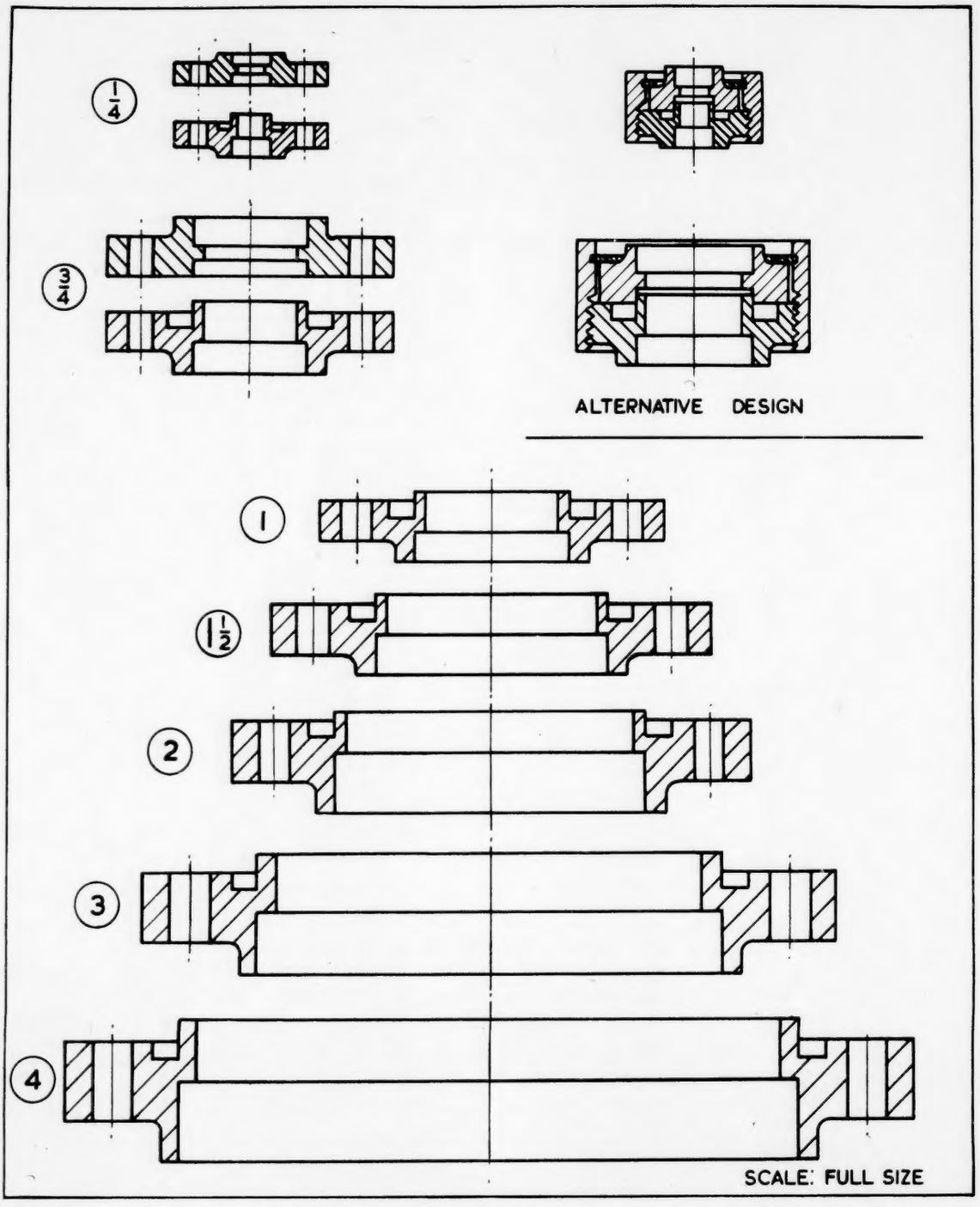
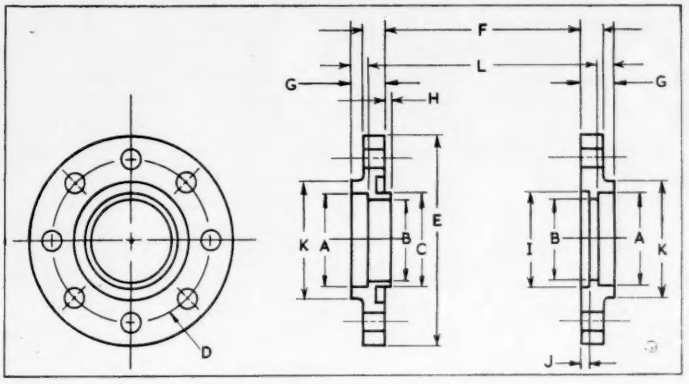


Fig. 3. Scale sections of several sizes of flanges with an alternative knurled-ring construction for the smaller sizes. All sizes are given in inches.

Vol
4
195



Size	*Bolts*		*Bolt Holes*		*"O" Ring*	*Groove*	*A*	*B*	*C*	*D*
	No. Off	*Size*	*No. Off*	*Size*						
$\frac{1}{4}$	4	*4	8	No. 32	$\frac{1}{4}\times\frac{1}{16}$	0.052 0.042 × 0.093	0.254 0.252	0.187	0.251 0.249	0.687
$\frac{1}{2}$	4	*6	8	No. 27	$\frac{1}{2}\times\frac{3}{32}$	0.083 0.073 × 0.128	0.504 0.502	0.406	0.501 0.499	1.125
1	4	*10	8	No. 8	$1\times\frac{1}{8}$	0.113 0.108 × 0.170	1.004 1.002	0.875	1.001 0.999	1.750
2	8	*10	8	No. 8	$2\times\frac{1}{8}$	,,	2.005 2.003	1.875	2.001 1.999	2.812
4	8	$\frac{1}{4}$	8	F	$4\times\frac{1}{8}$	,,	4.007 4.005	3.750	4.003 3.996	4.875
8	16	$\frac{1}{4}$	16	F	$8\times\frac{1}{8}$	,,	8.010 8.008	7.750	8.003 7.996	8.875
16	16	$\frac{3}{8}$	16	W	$\frac{1}{4}$	0.237 × 0.237 0.232 × 0.232	16.015 16.013	15.625	16.000	17.312
32	24	$\frac{1}{2}$	24	$\frac{17}{32}$	$\frac{1}{4}$	,,	32.025 32.020	31.500	32.000	33.562

Size	*E*	*F*	*G*	*H*	*I*	*J*	*K*	*L*
$\frac{1}{4}$	1	$\frac{1}{8}$	0.187	0.032	0.256 0.254	0.065	$\frac{7}{16}$	0.093
$\frac{1}{2}$	$1\frac{1}{2}$	$\frac{7}{32}$	0.312	0.047	0.506 0.504	0.078	$\frac{11}{16}$	0.172
1	$2\frac{1}{4}$	$\frac{1}{4}$	0.375	0.065	1.006 1.004	0.094	$1\frac{1}{4}$	0.187
2	$3\frac{3}{8}$	$\frac{3}{8}$	0.562	0.065	2.006 2.004	0.094	$2\frac{1}{4}$	0.375
4	$5\frac{1}{2}$	$\frac{1}{2}$	0.781	0.125	4.013 4.006	0.156	$4\frac{1}{4}$	0.500
8	$9\frac{1}{2}$	$\frac{5}{8}$	0.937	0.125	8.018 8.011	0.156	$8\frac{1}{4}$	0.656
16	$18\frac{1}{8}$	$\frac{7}{8}$	1.312	0.188	16.030	0.218	$16\frac{1}{2}$	0.844
32	$34\frac{5}{8}$	$1\frac{1}{8}$	1.687	0.250	32.050	0.281	$32\frac{1}{2}$	1.156

*American Screw No.

Table I. The dimensions under consideration for the final series of bolted flanges (see also opposite page). All dimensions are shown in inches.

minimum possible number of sizes and types of fittings. Otherwise, it would be too expensive to make and stock them.

(4) If it can be arranged easily, it would be convenient to be able to make simple connections between our standard fittings and metric fittings.

(5) Because of their fool-proof action and widespread availability, O-rings will be used to make the vacuum seal. (For ultra-low-pressure work another type of seal such as the knife-edge seal may well be adopted.)

(6) The bolted flanges should have eight bolt holes or some multiple of eight, in order that fittings may be assembled at the usual angles of 90° and 45°.

A study of the catalogues of metal jobbers has revealed that in all the above metals, tubing is available in even fractions of an inch in o.d. sizes with a very wide variety of wall thicknesses. Any size is available from the tubing manufacturers but that is of no significance since they will make up tubing only on very large orders. Steam-pipe sizes are available in fewer materials and usually only in two wall thicknesses, both of them quite heavy. Vacuum equipment needs only to support itself and 15 lb. sq. inch external pressure so that a light wall is preferable for most applications. Therefore, we are considering that the flanges should have sizes suitable for use with even o.d. sizes of tubing.

Fig. 1 is a line diagram showing a possible choice of sizes. In the upper circles are the o.d. sizes in inches of a ' first series ' of sizes from $\frac{1}{4}$ inch to 32 inch. It would be very desirable to limit the stock fittings to these sizes wherever possible. To fill the gaps a ' secondary series ' marked in the second set of circles could be permitted where such sizes were really needed. Below the line in Fig. 1 are two sets of metric sizes marked in millimetres. In the hexagons are the sizes suggested for standardisation in France by a technical committee of La Société Française des Ingénieurs Techniciens du Vide. In the circles is a series of sizes in even multiples of 5 mm. (except 12 mm.). Whether these sizes are widely available in Europe has not yet been determined, but, if so, it would be possible to have our standard fittings and the metric fittings chosen so that a simple adapting plate would serve to connect them.

Fig. 2 is a scale drawing of the pattern of bolted 1-inch flanges which is now under consideration by our committee. There is a male and a female flange. The male flange contains the O-ring and has a projection about equal to the wall thickness of the normal tubing. This projection fits into a slightly deeper recess in the female flange with a reasonably close radial clearance. The purpose of this projection is to provide alignment of the fittings independently of the bolt holes, so that the bolt holes may be well oversize and located to relatively loose tolerances. The bolt holes are the expensive part of bolted flanges so this keeps the costs down and makes the fittings easy to assemble while still giving precise location after assembly. There are eight bolts set no farther out than necessary to give good tool clearance. The hub on the back makes it simple to set the flange square on the tubing and keep it square while welding or brazing. The whole flange has been kept as light as possible consistent with a mechanical strength

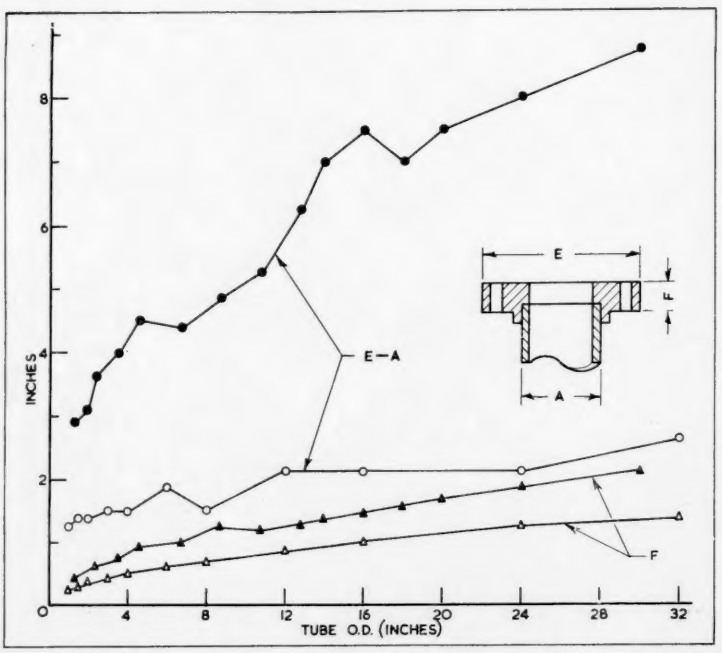


Fig. 4. Comparison of suggested vacuum flanges and 150-lb. steam-fitting flanges. Solid dots and triangles for steam-fitting, open circles and triangles for vacuum flanges.

Vol. 4 1954

comparable to that of the tubing itself.

Fig. 3 shows scale drawings of a few of these flanges to show the proportions in the various sizes. For the $\frac{1}{4}$ inch and $\frac{3}{4}$ inch size an alternative design is shown. These flanges are joined by a knurled ring having a snap-ring bearing on the back of the female flange. The knurled flange is cheaper than the bolted one since it can be made entirely on automatic screw machines.

Table I gives the dimensions under consideration for the ' first series ' of bolted flanges*.

Fig. 4 gives a comparison between this series of flanges and the 150-lb steam-fitting flanges which have often been used where space and weight were no consideration. The upper curve connecting the solid dots shows the diametrical overhang over the tubing diameter for the steam fittings. The second curve is the overhang of the flanges tabulated above. The two lower curves make a similar comparison between the thickness of the two types of flanges. There is much less weight (and thus cost) in the suggested flanges. Another point which is not so obvious is that in many applications, there is just not enough room between pieces of equipment mounted side-by-side to permit the use of steam-fitting flanges.

Comments both *pro and con* are earnestly solicited.

It is very much to the interest of experimental physicists that they make their comments while this work is at a preliminary stage so that their suggestions may be incorporated.

Yale University, W. G. WADEY.
New Haven, Connecticut,
United States of America.

20th March, 1956.

* A full table is available from the author.

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954

LETTERS TO THE EDITOR

A Mechanical Swirling Device for Vacuum Evaporation

Sommaire

DESCRIPTION EST DONNÉE d'un mécanisme qui communique un mouvement de rotation à un ou plusieurs flacons, simultanément. Ce système remplace le mécanisme à main employé auparavant dans la distillation sous vide de solvants volatiles, empêche les chocs et permet de distiller des produits à taux d'évaporation élevé.

IT IS OFTEN NECESSARY in chemical laboratories to free a solution from its solvent by evaporation and this operation is facilitated by the use of reduced pressure.

However, unless some precautions are taken, frothing and bumping will occur with resultant loss of material.

It is usual for the operator to swirl the evacuated flask of solution by hand in or over a steam-bath, so that local over-heating cannot take place and frothing is partly suppressed by centrifugal action. If the solution holds solvent tenaciously—as in the case of many oils—it may be necessary to swirl the flask manually all day to remove the last traces of solvent, even when very low pressures are employed. Clearly this procedure is tedious and time consuming.

On a small scale, mechanical stirring is not very satisfactory as it involves loss of solute to the stirring mechanisms and this may be a disadvantage where a dry residue is to be weighed in the flask after evaporation. Further, many of the vacuum sealing materials used for shaft seals are attacked by the common laboratory solvents.

The commercially available flask shakers, whilst producing very good turbulence in the contents of the flask, are not very suitable for solvent evaporation since they are likely to induce frothing under vacuum conditions.

Hand swirling, on the other hand, produces a vortex in the flask contents which has a large surface area and by its centrifugal action tends to suppress foaming. This is the normal laboratory method and occupies one operator per flask during the period of evaporation.

We have arranged a simple mechanism which moves the flask in a manner similar to that produced by wrist action and so dispenses with any internal rotating parts. By driving from above the level of the flask, we leave the bottom of the flask itself clear, so that it may be held in or over the source of heat whilst evaporation is taking place.

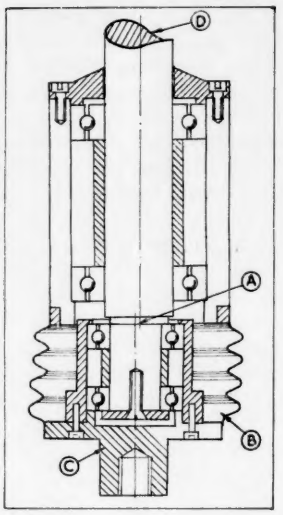

Fig. 1. A detailed drawing of the drive unit.
Legend: A.—Off-centre, off-axis spigot, producing a conical orbit. A cylindrical orbit is produced if this portion of the shaft is turned parallel with the upper section. B.—Metal bellows which keeps the mechanism clean and prevents rotation of the drive boss. C.—Drive boss. D.—To geared motor drive.

The action is identical with that produced by hand swirling, but being mechanical it is more regular and can be adapted to greater rates of evaporation by increasing the speed of swirling. Moreover, one drive may be used to swirl a number of flasks at once, so that one operator can attend to four or six flasks at a time.

The mechanism of the drive unit is as follows: A shaft is rotated in ball-races by means of a variable-speed geared motor. One end of this shaft projects from the ball-race housing and has an eccentric spigot, turned parallel to the main shaft but displaced from it by a small distance. On this spigot is mounted a second ball-race housing of a similar diameter to that in which the first shaft runs and the two housings are connected with a length of metallic bellows tubing, (see Fig. 1). The bellows serves the double purpose of preventing the revolution of one housing with respect to the other and at the same time excluding corrosive vapours from the bearings.

When the drive shaft is rotated, the lower ball-race

Vol.
4
1954

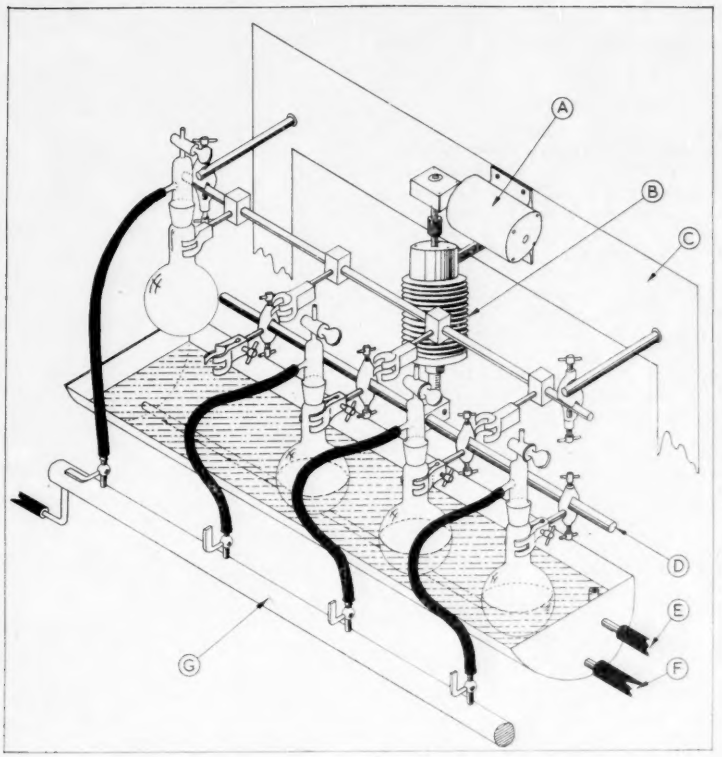

Fig. 2. A view of the complete assembly of the swirling device. The flask in the upper left corner is parked in the upper clamp after completion of the solvent removal.

Legend:
A–Geared Motor
B–Drive Unit
C–Support Framework
D–Driving Rod
E–Overflow
F–Steam Inlet
G–Vacuum Manifold.

housing describes small circles but remains in a constant orientation. Consequently any apparatus connected to the lower housing will also describe small circles and simulate wrist action or swirling.

Such a drive unit is in use in these laboratories for routine evaporation of light solvent from up to six flasks and is shown in Fig. 2.

This drive unit has $\frac{1}{2}$-inch shafts at each end, and is driven by a variable speed, $\frac{1}{20}$ h.p.. geared motor with a maximum speed of 250 r.p.m. A length of $\frac{1}{2}$-inch diameter, alloy rod is fixed horizontally to the drive end of the unit and carries standard laboratory clamps. A trough shaped steam bath is arranged underneath and in front of this is situated a vacuum manifold with cocks at intervals. Above the drive unit is another row of clamps into which the flasks may be placed one by one as they reach dryness, so that overheating of the residues is avoided. As the amplitude is small, it is easy to remove flasks from the clamps whilst the system is in motion.

For 250-ml. flasks an amplitude of about $\frac{1}{8}$-inch to $\frac{3}{16}$-inch is quite sufficient and control is obtained by varying the speed of swirling.

Once made, the unit itself can also be used for agitating titration flasks, for hydrogenations and for similar laboratory duties.

For small flasks up to 250 ml. the motion may be transmitted directly through a standard cone connection fitting into the neck of the flask and, where this method is applied, any number of flasks can be swirled and pumped from common pumping and driving systems.

A modification of this device can be used to provide a flexible wide-bore link between a swirling distillation flask and a static glass fractionating and condensing assembly. This takes the place of the conventional air or nitrogen leak in vacuum distillations, where recovery of both distillate and residue is important.

This work is the subject of a patent application.

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1st January, 1956.

Some Aspects of Micro-Moisture Determination

Sommaire

UNE DESCRIPTION est donnée d'une méthode absolue de la détermination de la teneur en eau. L'échantillon est mis sous vide dans un système clos, et, l'augmentation de pression, due à l'évaporation de la vapeur d'eau contenue, fait dévier un des bras d'un manomètre à huile et est mesurée sur une échelle calibrée de 5 à 120 microgrammes. Cette méthode a été employée pour examiner : l'effet de la température sur le taux de séchage d'un matériel pendant les dernières périodes de déshydration sous vide ; le taux d'absorption de matériaux lyophilisés ; la diffusion de l'eau au travers de differents types de bouchons de caoutchouc pour trouver celui qui convient le mieux à la fermeture étanche de bouteilles contenant des produits lyophilisés ; le taux de séchage sous vide des plastiques et l'efficacité de produits déshydratants pour determiner la relation entre la vapeur d'eau absorbée et la pression de la vapeur d'eau résultante.

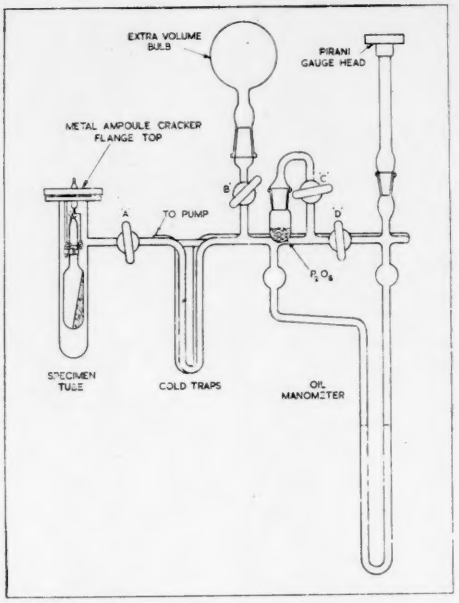
Introduction

During the past few years, the measurement of the moisture content of various materials, particularly freeze-dried biological and pharmaceutical preparations, has become increasingly important. This is so because the storage life of such dried substances is adversely affected by a high moisture content. There is also evidence to show that with some substances overdrying can be harmful.

A very necessary condition for any method of moisture determination of complex materials is that it should be possible to differentiate between free residual moisture and chemically ' bound ' moisture. Adaptation of a technique devised by Vincent and Simmons[1] led to the development of an absolute method satisfying the above condition. The use of this apparatus has revealed a number of interesting results and wider applications than those originally envisaged.

Description of the Methods

Method I. The method provides for the evacuation of the apparatus to a pressure of 0.02 mm. Hg. The sealed sample in the specimen tube is cracked open under vacuum by a heated Nichrome wire loop and the top of the tube is displaced by tapping it with a plunger operated by an external magnet. The sample is then exposed to the cold trap for a short time and (with all taps closed, so isolating a known volume) the moisture thus condensed is re-evaporated and measured by exposing it to the oil manometer. After further measurements, the total moisture content is obtained.



The moisture determination apparatus used in the experiments.

Some examples of freeze-dried vaccines, weighing below 0.1 g. and containing less than 0.5% (by dry weight) residual moisture, which have been examined, for moisture content in the apparatus are :—

Yellow Fever Vaccine, Sheep-Pox Lymph, Brucella Abortus, Newcastle Disease and Caprinised Rinderpest Vaccine.

Method II. By varying the above method the relationship between the moisture content of the sample and its equilibrium vapour pressure is initially found and thereafter a single vapour pressure measurement suffices to determine the moisture contents of any further sample of identical composition. The sample is calibrated by measuring its equilibrium vapour pressure and exposing it to one arm of the oil manometer after the successive removal of measured quantities of water.

Using a special technique, the lowest vapour pressure measured so far with the method is 0.0014 mm. Hg. This is obtained by exposing the sample to a larger volume (by the introduction of a bulb in the apparatus) and when equilibrium is reached, after isolating the sample, condensing the water vapour which is subsequently expanded into the original small volume. The reading thus obtained is divided by the ratio of the volumes A : (A+B), where A= volume of apparatus and B=volume of bulb.

Vol. 4 195

Example of Method II. A vapour pressure/moisture content curve was obtained for a sample of blood plasma (see Fig. 1). This curve enabled the moisture contents of other plasma samples to be found from a single equilibrium vapour pressure reading. The curve gives, at the same time, the highest permissible condenser temperature for efficient final drying of blood plasma, *i.e.* the vapour pressure of the sample of blood plasma with 0.4% residual moisture content was 0.02 mm. Hg which corresponds to the vapour pressure of ice at −54°C. The condenser temperature must therefore be below −54°C in order to complete the drying of the blood plasma at room temperature to a moisture content of 0.4%.

It may also be seen from Fig. 1 that there is an hysteresis loop between the absorption and desorption curves. This loop has not yet been explained.

Tests and Results Obtained by Method I

The following are typical examples of the kind of information which use of the apparatus can provide by employing Method I.

The Effect of Temperature on Freeze-Dried Materials. In freeze drying, the material is dried in two stages. Primary drying removes the water of suspension and about 98% of the water contained in the material, usually by condensing it on a refrigerated surface. In secondary drying, the material is transferred to a separate system, where it is dried over phosphorus pentoxide to a moisture content in the region of 0.5% and subsequently hermetically sealed. As secondary drying is somewhat lengthy it is often impractical and uneconomic to carry it out in the primary chamber.

For some substances the secondary drying process may be shortened by raising their temperature to a point just below that at which decomposition occurs. The moisture determination apparatus described above has been successfully used to find the temperature levels at which chemically combined moisture is released from a substance under vacuum.

Blood plasma was taken as typical of a range of biological substances (citrated blood consisting of 420 ml. blood, 100 ml. 2% disodium citrate, 20 ml. 15% dextrose). A number of primary freeze-dried blood plasma samples, each with similar moisture contents, were tested consecutively for 50 hours at fixed temperatures between 0°C and 100°C. The samples were heated by a constant-temperature oil bath surrounding the specimen tube.

From the results (see Figs. 2 and 3) it was found that the amount of moisture evolved after 6 hours varied linearly with change in temperature from 0°C to 60°C. Above 60°C the amount continued to vary linearly with temperature but at an increased rate.

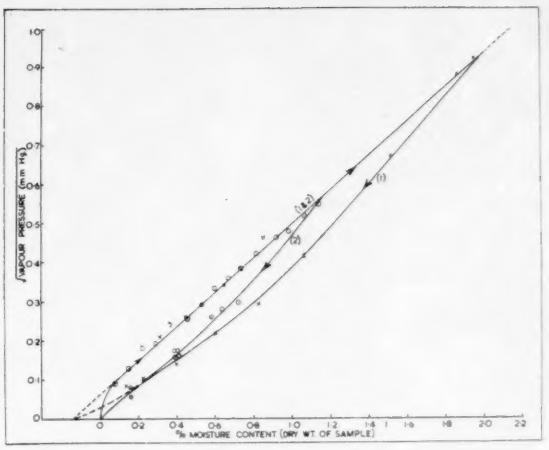


Fig. 1. Vapour pressure curves of freeze-dried blood plasma. The same sample was used throughout these tests. Dry weight: 0.156 g. *Key:* ° = First test, Dot in° = Second test, → = Adsorption at 24°C, ← = Desorption at 24°C.

Also, the amount of moisture evolved after 50 hours was fairly constant at temperatures between ambient and 60°C. Above 60°C there was a further rapid evolution of moisture, indicating decomposition.

It was also possible from these tests to find the times necessary to moisture-determine a freeze-dried blood plasma sample at various temperatures as shown in Table I.

Table 1

Temperature of Freeze-Dried Sample °C.	*Times Required to Remove all Residual Moisture* hrs.
0	>50
23	50
45	25
60	6

Generally the time necessary for a moisture determination is almost impossible to predict as it depends greatly on the kind of material and the temperature of drying. Figs. 4 and 5 show that a freeze-dried sample (weight 0.4 g.) of 16% glucose and 3% gelatine took 500 hours (5 weeks) to test at room temperature, after being freeze-dried for 66½ hours, whereas the test made on a powdered sample of hormone substance (also at room temperature) was completed in 35 minutes. Another factor influencing the time, is the shape of the sample. Table II gives the times taken up by moisture determination tests for a range of typical materials.

Table II

Material	*Form of Sample*	*Remarks* (*Measurements in inches*)	*Dry Weight of Sample*	*Duration of Test at Room Temperatures* Moisture 90%	Removed All
			g.	*hrs.*	*hrs.*
Freeze Dried Materials					
Yellow Fever Vaccine (Infected Chick Embryo)	Slant Wedge Shape in Ampoule		0.04	25	50
Rinderpest Goat Tissue Vaccine	,,		0.286	21	50
Newcastle Disease Vaccine	,,		0.023	25	50
Brucella Abortus Vaccine :					
(1) Suspension in Smith Naylor Medium	,,		0.07	8	25
(2) Suspension in Agglutinating Serum	,,		0.092	19	30
(3) Suspension in Agglutinating Serum	,,	Material Fragmented	0.092	3	25
Blood Plasma	,,		0.15	25	50
Sheep-Pox Lymph :					
(1) Digest Broth and Vaccinia Virus	,,	Frothy Appearance	0.017	1.3	3
(2) 1% Peptone and Vaccinia Virus	,,		0.015	0.5	1
Bovine Serum (3 Parts Serum, 30% Glucose Meat Broth)	,,	Frothy Appearance	0.008	3	8
Glucose (16% Glucose, 3% Gelatine)	,,		0.403	300	500
Artery Tissue (Dried at −30°C for 7 Days)	—		0.046	2	50
Miscellaneous Materials					
Confectionery Glucose	Powder		1.4	25	50
Soap	,,		0.05	0.5	8
Hormone Substance	,,		0.035	0.1	0.6
Plastics					
(1) Cellulose Acetate	Block	0.112 × 0.224 × 0.448	0.2	70	100
(2) Cellulose Acetate (Water Immersed 68 hrs.)	,,	,,	0.2	15	100
(3) Perspex (Water Immersed 68 hrs.)	,,	,,	0.19	45	80
(4) Nylon	Chips	Average Size: 0.125[8]	0.2	45	80
(5) Perlon	Foil	Thickness 0.005	0.2	0.5	70
Kraft Paper	Tissue	Thickness 0.0003	0.16	4	50
Natural Rubber	Disc	Diameter 0.75 Thickness 0.125	1.55	34	55

Vol 4 195

Moisture Re-Absorption. Freeze-dried samples, being easily reconstituted, are hygroscopic but if the rate at which they take up moisture from the atmosphere is known, the maximum safe time available for sealing the samples from the atmosphere can be calculated. To obtain this information a re-absorption test was made on a stored freeze-dried sample of adrenal cortotrophic hormone (ACTH). The initial moisture content of the sample was 6.1%, (weight 0.015 g.). After exposing it to an atmosphere of 100% relative humidity for 50 minutes, the moisture content rose to 20.3%, *i.e.* three times the former value.

Water Vapour Diffusion Through Rubber. As it is known that moisture diffuses through rubber,[2,3] tests were carried out at room temperature in the moisture determination apparatus to determine the rate of moisture diffusion through natural and other grades of rubber stopper closures for small bottles containing freeze-dried material. This proved to be high (enough to hydrate a 1 g. sample 10% by weight in 1 year at 100% relative humidity). Samples of different rubbers were then obtained and tested to find a more suitable seal. Previous work on the subject was reported by Flosdorf and Webster in 1938[4] and was further discussed by Flosdorf in 1949[5].

It was found that butyl rubber had a moisture diffusion rate 10 to 20 times lower than the rate for wax impregnated natural rubber and neoprene. There are many cases in vacuum work where moisture cannot be tolerated and it is necessary to realize that rubber ' O ' rings and gaskets can be a source of water vapour diffusion into a vacuum system, although this is not significant in continuously pumped systems, see Table III.

Table III*

Stoppers	*Permeability Rate*
Red, Wax Impregnated, Natural Rubber	7.6×10^{-9}
White, Wax Impregnated, Natural Rubber	5.3×10^{-9}
Natural Rubber Used for Penicillin Bottles	(1) 8.2×10^{-9} (2) 4.1×10^{-8}
Slab Rubber	
Gaco Rubber (Neoprene) (Shore Hardness E.75)	5.8×10^{-9}
Butadiene (Acrylonitrile Type)	2.5×10^{-8}
Butyl Rubber	4.4×10^{-10}
Wax Impregnated Natural Rubber	6.4×10^{-9}

* This table gives the results of the permeability tests of the rate of water vapour in c.c/sec. at N.T.P. which diffuses through 1 sq. cm. of material, 1 cm. thick, when the pressure difference is 1 atmosphere.

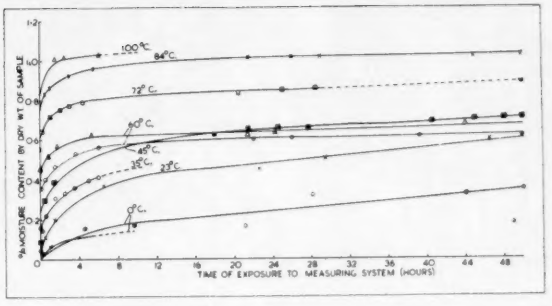
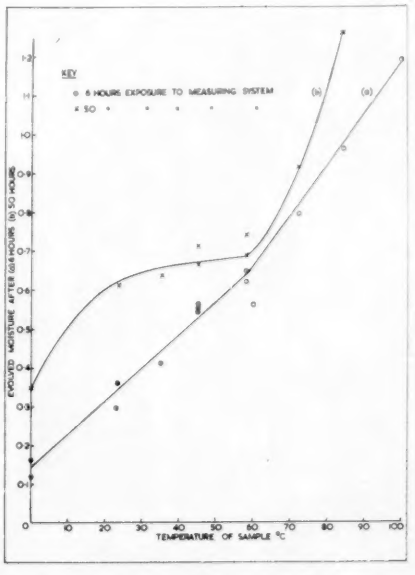


Fig. 2. Evolution of moisture from freeze-dried blood plasma at different temperatures.

Drying of Plastics to be Metallised. Metallic finishes to plastic mouldings are now commonly achieved by the vacuum evaporation technique which requires a vacuum of around 5×10^{-4} mm. Hg to be maintained. Plastics contain many volatiles which are liberated in a vacuum. The manner in which one of them, water vapour, evaporates determines the design of the pumping system for the metallising unit. In some

Fig. 3. Effects of temperature on loss of moisture from freeze-dried blood plasma.



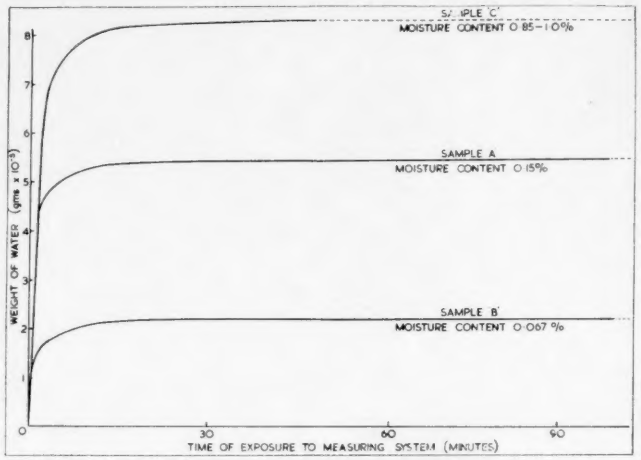


Fig. 5

Fig. 5. Evolution of moisture from powdered hormone substances.

Fig. 4. Evolution of moisture from glucose (16%)—gelatine (3%) sample freeze-dried for 66½ hours. Dry weight of sample: 0.403 g. Average temperature during test: 16°C. Moisture content: 2.2% (by dry weight).

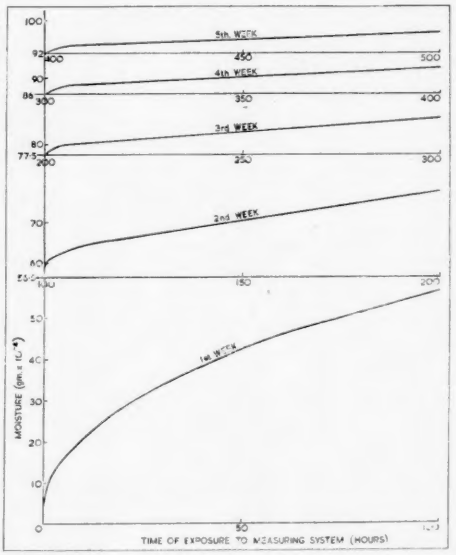


Fig. 4

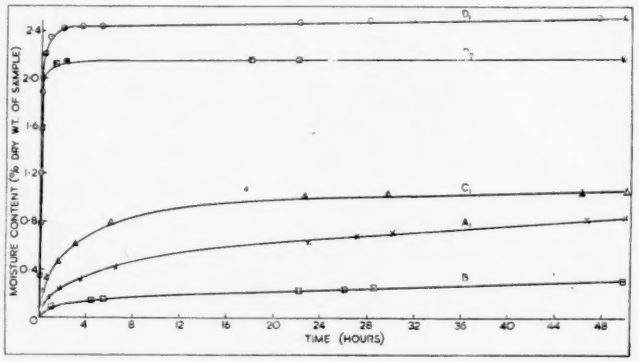


Fig. 6

Fig. 6. Rates of drying for plastics. A_1—Cellulose acetate block (mineral-filled). B—Perspex block (immersed in water for 68 hours at 20°C). C_1—Nylon chips. D_1—Perlon foil. D_2—Perlon foil (heated to 55°C).

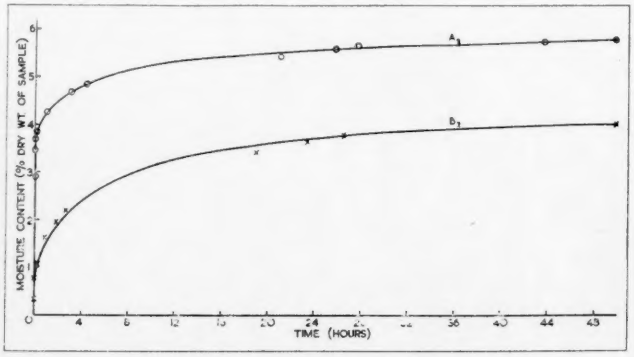


Fig. 7

Fig. 7. Rates of drying for plastics. A_2—Cellulose acetate block (mineral-filled and immersed in water for 68 hours at 20°C). C_2—Nylon chips (immersed in water for 42 hours at 20°C).

cases the moisture is readily liberated and would contaminate the sealing oil of the mechanical pump, whilst in other cases it is removed during the later stages of evacuation by the diffusion pump.

Tests have been made in the apparatus to determine the rates and quantities of moisture liberated from samples of cellulose acetate block, perspex block, nylon chips and perlon foil[6]. The required data was obtained from Figs. 6 and 7 plotted from the results of the tests.

Evaluation of Desiccant Efficiency. To determine the efficiency of a desiccant *in vacuo*, it is necessary to know the amount of water it will absorb, before exerting the highest permissible vapour pressure. This information has been found from vapour pressure tests on silica gel and activated alumina. It can be seen from Fig. 8 that activated alumina is a better desiccant than silica gel below pressures of 8 mm. Hg. The maximum amount of water that it will absorb before it exceeds a vapour pressure of 0.02 mm. Hg, is 2% of its own weight.

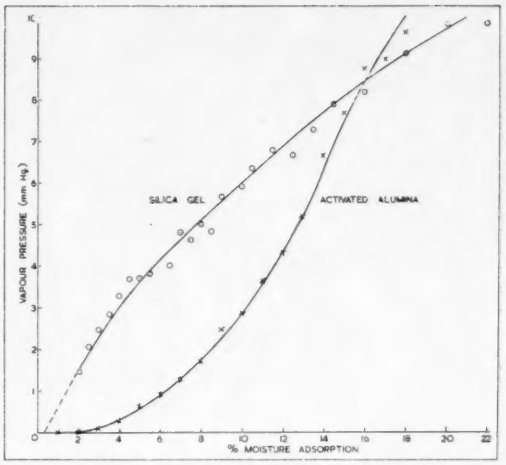


Fig. 8. Vapour pressure curves of silica gel and activated alumina on water adsorption at 24°C.

Method I Compared with an Oven Method

Comparative tests have been made with a vacuum oven method of moisture determination[7]. The operational procedure for the vacuum oven method is as follows :—A freeze-dried sample is placed in an open metal foil cup and weighed at certain time intervals so allowing it to absorb moisture from the atmosphere. The rate of moisture absorption is then known, and, assuming linearity, the weight of the sample after drying can be determined. The sample is then placed in the vacuum oven, at a temperature of 50°C, for a pre-determined length of time, then after a further weighing, the loss in weight and therefore the moisture content of the sample, can be found.

The materials used for the tests were samples of freeze-dried Yellow Fever vaccine and distemper vaccine. The latter samples were in three batches. Batch 1 was taken at random from the freeze-dried material, batch 2 was exposed to the atmosphere for a maximum of 5 minutes prior to being sealed and batch 3 was exposed to the atmos-

Table IV

Apparatus	*Sample*	*Av. Temp. of Sample during Test* °C	*Moisture Content* %	*Moisture Determ. Time* Mins.
Method I	Yellow Fever Vaccine	50	0.21	45
Vacuum Oven Method	,,	,,	0.20	45
	,,	,,	0.25	45
Method I	Canine Distemper Vaccine *Batch* 1	50	0.75	90
Vacuum Oven Method	,,	,,	1.0	60
	,,	,,	1.1	60
Method I	Canine Distemper Vaccine *Batch* 2	50	0.4	120
Vacuum Oven Method	,,	,,	0.46	60
	,,	,,	0.45	60
Method I	Canine Distemper Vaccine *Batch* 3	50	0.9	110
,,	,,	60	0.85	60
,, (Large-bore tube connecting specimen tube to cold trap)	,,	50	1.12	70
Vacuum Oven Method	,,	50	1.37	60
	,,	,,	1.4	60

phere for 1 hour. For the results of the tests see Table IV.

Although the results suggest that the two methods employed were equally efficient, it must be remembered that the sensitivity of the vacuum oven method relies largely on the sensitivity of the available weighing balance and on the accuracy of the operator. Method I on the other hand is an absolute method and can be read to a sensitivity of 1×10^{-6} g. Also by taking only one measurement of the weight of moisture removed, the vacuum oven gives no indication of decomposition occurring in the sample. Method I however, will indicate decomposition during a test by a plateau in the drying curve.

Conclusions

Experience with the apparatus has shown that it can fulfil the need for a method of directly determining moisture content of thermo-labile and other materials which hitherto has had to be carried out by methods which are laborious and not reliable. In addition, it has been found particularly valuable for a variety of tests such as:—The effect of temperature on the decomposition of samples in a vacuum, membrane diffusion properties, rates of water evolution for determining vacuum equipment pumping requirements and vapour pressure measurements for assessing desiccant efficiency.

My thanks are due to Mr. T. W. G. Rowe for encouragement in this work and to Mr. A. S. D. Barrett, Technical Director of Edwards High Vacuum Ltd., for permission to publish this letter.

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25th January, 1956.

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Some Remarks on Ion Pumps

Sommaire

LE PROCÉDÉ DE POMPAGE des pompes à transport ionique ou des pompes getter ionique est brièvement discuté; il dépends de la distance anode-cathode, de la tension, de la densité du courant, de la surface de la cathode et du type de gaz.

IN RECENT YEARS numerous papers appeared dealing with the methods producing a vacuum, based on ionisation phenomena. The primary process of such a pumping action is the ionisation of gas molecules or atoms, and imparting them with a preferred direction by an electric field. The secondary process is the removal of the gas ions from the pump. The various methods developed differ with regard to the removal of the ions from the pumping area. With this point in mind recent papers on the subject can be classified into two groups.

(1) The movement of the ions produced a pressure difference in the system, and the ions, after loosing their charges, are removed continuously by a backing pump. This system may be called *ion-transport pump.*[1-4]

(2) In the other group of ion pumps, the ions are directed to a surface in the system, where they are adsorbed in some form preventing them from returning into the pumping area. Here the final removal of the gas is based on the physical and chemical properties of the materials used and this system may be called *ion-getter pump.*[5-9]

Re. ion-transport pump. The dependence of the pumping speed on the geometry and other characteristics of the pump was discussed in an earlier paper, based on an actual model of the pump.[4]

Re. ion-getter pump. Von Engel[10] and Alpert[5] have calculated the pumping speed of that pump. Both authors used the same simplifications. They supposed, that every ion originated in the process will be captured on the cathode surface, and not return to the pumping area. These simplifications are questionable in view of experiments of Bloomer and Haine[6], who called attention to the desorption and back diffusion of molecules from the cathode surface.

Taking these phenomena into consideration, the condition of pumping is given by :

$$P_i > N_d \qquad (1)$$

where P_i denotes the average number of activated

molecules in unit time near the surface, and by N_d the average number of desorbed molecules.

The pumping speed can be described by the number of particles finally removed :

$$S = -F/P_i - N_d/\frac{c}{p} \qquad (2)$$

In this formula F denotes the effective cathode surface, p the pressure and c a constant. The determination of P_i and N_d is difficult because of the uncertainty of the number of activated particles generated in the vicinity of the cathode surface. The nature of the gas and that of the active surface must also be taken into consideration.

Conclusion

Cathode-Anode Distance. Equ. (2) shows that contrary to ion-transport pumps, which require a large cathode-anode distance and a small voltage (100-1,000 V), the ion-getter pumps need small cathode-anode distance and high accelerating voltages (1,000-10,000 V).

Ion Current Density. There is another appreciable difference between the two pumping mechanisms as a result of the ion current density requirements. The efficiency of an ion-transport pump can be improved by increasing the ion current density. The ion-getter pump requires only a small ion-current density for its operation but its efficiency can be increased by enlarging the effective cathode surface.

Type of Gas. Another important question is the effect of the nature of the gas on the pumping speed of the two types of pumps. In the case of an ion-transport pump the pumping speed for nitrogen is theoretically 10 times larger than that for a noble gas. In the case of an ion-getter pump this factor is about 1,000.

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BOOK REVIEWS

Optical Properties of Thin Solid Films. By O. S. Heavens. (Butterworths Scientific Publications, London, 1954.) Pp. 261. Price £1 15s. 0d.

A layer of material whose thickness is comparable with the wavelength of visible light (0.5μ), so that it may exhibit interference phenomena, is conventionally known as a ' thin ' film. The theoretical basis for a study of the properties of such films has existed since the last century, but it is only during the past twenty years that developments in vacuum technique have made it relatively easy to fabricate a large variety of materials in the form of uniform thin films. Many people are familiar with the ' bloomed ' lens, which is a very simple application of thin film technique, but the remarkable properties of the more complex assemblies of thin films are often little known even in the scientific world. The published information on thin films is widely scattered throughout a number of journals, and workers in this field will be grateful to Dr. Heavens for collecting it together in this authoritative survey. The introduction provides a brief

survey of the scope of the book, which describes in logical order the production, physical structure, optical properties and most important uses of thin films.

The chapter devoted to the formation of thin films is concerned mainly with the method of thermal evaporation, which is the most widely used technique at the present time. After an outline of the general precautions which are essential for successful evaporation there are descriptions of several of the more common forms of evaporator, together with a list of the materials for which each is most suitable. The methods of sputtering, electrolytic deposition, and chemical reaction are briefly mentioned and finally, the more fundamental aspects of the condensation process are adequately dealt with.

Dr. Heavens devotes the next chapter to a discussion of the knowledge of film structure which has been obtained by the use of optical microscopy, electron microscopy and electron diffraction. The available experimental evidence suggests that a very thin metallic film consists of a large number of partially independent droplets, whose size and state of aggregation depend upon the rate at which the film was formed and also upon its subsequent heat treatment. However, as the thickness of the film is increased it becomes a closer approximation to the theoretical concept of a plane, parallel-sided layer, to which Maxwell's electromagnetic equations may be applied.

The detailed application of this classical theory forms what is probably the most useful section of Dr. Heavens' book. Some half a dozen different methods of performing numerical calculations on thin films have been collected together and reduced to a common notation, which greatly simplifies the task of selecting the most suitable procedure for any particular problem. After deriving the basic theoretical equations for the reflection and transmission of light at a solid interface, the author describes methods involving addition of amplitudes, vector summation, the use of matrices, the transmission line analogy and the use of graphical methods. In each case the method is illustrated by applying it to a practical example and anyone who has attempted to carry out computations involving two or more films will sympathise with Dr. Heavens' use of phrases such as ' miserably complicated ' and ' excessively cumbersome '. The choice of a suitable notation is a problem in itself and the use of unwieldy symbols such as E_{np}^{+} is virtually unavoidable. The printers are to be congratulated upon the way in which they have handled the many complex algebraic equations, although the reviewer was slightly irritated by the indiscriminate use of the alternative forms ϵ^{x_ψ} and $\exp(\chi_\psi)$ for the exponential function. Readers should note that in figure 4.17 the value of n_2d_2 is given incorrectly, and it is difficult to see how the author arrives at the numerical results in table 4.1 by starting from the data quoted in the text.

The practical problems of measuring film thicknesses and optical constants provide the subject matter for the longest chapter in the book. Some thirty different mechanical, electrical and optical methods are described and critically compared, and ample references are given for further reading. Most of the mechanical and electrical methods merely determine the mass per unit area, and, since the bulk density can seldom be employed with confidence, this yields only an approximate value for the film thickness. The optical methods, based on interference phenomena, yield results of much greater accuracy (better than $\pm$ 1%) and between them they cover film thicknesses ranging from tens of Ångström units to a few microns. The standard of the diagrams in this chapter, and indeed throughout the book, is to be greatly commended. In all cases they convey their information clearly and concisely and include no irrelevant details.

The results of measurements made on thin films by a number of different workers show a perturbing lack of mutual agreement, although this can largely be attributed to the differing conditions under which the films were formed. The results for silver and gold are reproduced in detail and it is shown that the optical constants of absorbing films are extremely structure sensitive, often differing very appreciably from the bulk values. This of course makes it difficult to confirm theoretical predictions, but most of the changes which take place as the film thickness is increased may be plausibly explained by the theory published fifty years ago by Maxwell Garnett, and later developed more fully by Schopper.

The concluding chapter provides a brief survey of the applications of thin films as anti-reflection coatings, high efficiency mirrors, polarisers and narrow band-pass filters. Descriptions of the various methods of monitoring film thicknesses are also included.

Whilst not being an exhaustive treatise, this book does fulfil the author's claim ' to give a representative picture both of the potentialities and the limitations of thin films in relation to optical studies '. The two hundred references provide a useful lead for those requiring more detailed information and it will undoubtedly become a standard reference work to be found in any laboratory which has facilities for producing thin films.

K. H. JOLLIFFE.

. . . of vacuum interest

Research Progress

Student Theses

Base Pressure Studies in Rarified Supersonic Flows.
Author : *L. L. Kavanau.*
Degree : Ph.D.
Completed : November 1954.
Institute : Dept. Mech. Engng., University of California, Berkeley, U.S.A.

Design of a Two-Compound Microbalance for Low Density Wind Tunnels.
Author : *R. N. Latz.*
Degree : M.S.
Completed : July 1954.
Institute : Dept. Mech. Engng., University of California, Berkeley, U.S.A.

Lyophilisation and Its Application in Microbiology.
Author : *A. Rico.*
Degree : M.D. (Vet.).
Completed : 1954.
Institute : University of Paris, France.

The Leak Problem in Vacuum Systems. Experimental Set-Up of a Mass-Spectrometer Leak Detector.
Author : *R. Geller.*
Degree : D.Sc.
Completed : November 1954.
Institute : University of Paris, France.

Experimental Study of the Electrical Conductivity of Complex Metal-Dielectric Deposits Obtained by Evaporation.
Author : *C. Feldman.*
Degree : Doctorate.
Completed : 1955.
Institute : University of Paris, France.

Research Projects

(in Progress during 1955)

Cooling Tower Installation for Low Density Wind Tunnel.
Worker : *L. Talbot.*
Institute : Dept. Mech. Engng., University of California, Berkeley, U.S.A.

Indication: Investigation into the problem of increasing the cooling capacity of 5-stage steam ejector pumps employed in the equipment.

Low Pressures.
Workers : *S. A. Schaaf and L. Talbot.*
Sponsor : Office of Naval Research.
Institute : Dept. Mech. Engng., University of California, Berkeley, U.S.A.

Indication: Low pressure phenomena in fluid flow occurring between 10 mm. and 10^{-3} mm. Hg have been investigated.

Fundamental Study of Metal-Ceramic Bonding.
Worker : *J. A. Pask.*
Sponsor : U.S. Atomic Energy Commission.
Institute : Dept. Mech. Engng., University of California, Berkeley, U.S.A.

Indication: The wettability of metal on glass is investigated and a specially developed vacuum furnace is described which operates at 10^{-6} mm. Hg.

Low Pressure Drop Vacuum Still.
Institute : Bureau Engng. Res., University of Alabama, U.S.A.

Low Temperature Pumpability.
Institute : Inst. Engng. Res., University of California, Berkeley, U.S.A.

The Determination of the Feasibility of a New Method of Deairing Clay and of Adding Moisture under Vacuum Prior to Extrusion.
Institute : Coll. Engng., University of California, Los Angeles, U.S.A.

Flow of Gases Through Nozzles.
Institute : Engng. Expl. Station, University of Colorado, U.S.A.

High Vacuum Metallising.
Institute : Coll. Engng., University of Detroit, U.S.A.

Study of Thin Films Deposited on Methyl Methacrylate.
Institute : Dept. Engng. Res., North Carolina State College of Agriculture and Engineering of the University of North Carolina, U.S.A.

Vacuum Casting of Titanium Alloys.
Institute : School Engng., University of Southern California, U.S.A.

Vapour Liquid Equilibria of Petroleum Oils under Vacuum.
Institute : Bureau Engng. Res., University of Texas, U.S.A.

Rotary Vacuum Filtration for Removal of Algae from Liquid Substrate.
Institute : Bureau Engng. Res., University of Texas, U.S.A.

Lectures

Sweden. KUNGL. TEKNISKA HOGSKOLAN, FYSIKALISKA INSTITUTIONEN, STOCKHOLM 70 (Technical University of Stockholm, Physics Dept.) has organised a course of 6 lectures on the subject of OPTICS OF THIN FILMS, during the period 13th January-17th February 1956. The course consisted of the following lectures :—

Jan. 13th Preparation of Optical Thin Layers and Their Physical Properties.
Lecturer : *E. Ingelstam.*

Jan. 20th The Diffusion of Light in Thin Layers I.
Lecturer : *E. Djurle.*

Jan. 27th The Diffusion of Light in Thin Layers II.
Lecturer : *E. Djurle.*

Feb. 3rd Interference Filters.
Lecturer : *L. O. Hendeberg.*

Feb. 10th Technical Manufacture and Control of Thin Layers.
Lecturer : *L. Johansson.*

Feb. 17th Polarisation Effects in Thin Layers and Their Use for Measurement Purposes.
Lecturer : *L. Johansson and E. Ingelstam.*

●

United States. THE INTERNATIONAL COMMISSION FOR OPTICS has organised a symposium to take place at the Massachusetts Institute of Technology, Cambridge, Mass., from the 28th March-3rd April 1956 which will be devoted to FRONTIERS IN PHYSICAL OPTICS. Among other groups of subjects there will be lectures and discussions on THIN FILMS AND FILTERS. Further information can be obtained from Dr. S. S. Ballard, Visibility Laboratory, Scripps Institution of Oceanography, University of California, San Diego 52, Calif, U.S.A.

●

United Kingdom. THE INSTITUTION OF MINING AND METALLURGY, 44 PORTLAND PLACE, LONDON, W.1, organised a symposium on THE EXTRACTION METALLURGY OF SOME OF THE LESS COMMON METALS, which was held at the Royal Society of Arts, London, W.C.2, from the 22nd-23rd March 1956. 22 lectures were presented of which the following 7 had a bearing on vacuum metallurgy :—

Factors Influencing the Magnesium Reduction of Uranium Tetrafluoride.
Presented by : *J. Harper and A. E. Williams.*
U.K. Atomic Energy Authority, Springfields.

Processes for the Extraction of Vanadium.
Presented by : *C. Tyzack and P. G. England.*
U.K. Atomic Energy Authority, Warrington.

The Production of High-Purity Vanadium Metal.
Presented by : *E. Foley, M. Ward and A. L. Hock.*
Magnesium Elektron Ltd., Manchester.

The Selection of a Process for Development for the Production of Pure Niobium.
Presented by : *G. K. Dickson and J. A. Dukes.*
U.K. Atomic Energy Authority, Warrington.

The Extraction of Pure Niobium by a Chlorination Process.
Presented by : *A. B. McIntosh and J. S. Broadley.*
U.K. Atomic Energy Authority, Warrington.

The Preparation of Beryllium Metal by Thermal Reduction of the Fluoride.
Presented by : *D. A. Temple and L. J. Dereham.*
Imperial Smelting Corp., Ltd., Avonmouth.

Purification of Metals by Intermediate Formation of Their Stable Halide Vapours with Application to Beryllium and Titanium.
Presented by : *P. Gross and D. L. Levi.*
Fulmer Research Institute, Stoke Poges, Bucks.

●

France. LA SOCIETE DE RADIOELECTRIQUE and LA SOCIETE FRANCAISE DES INGENIEURS TECHNICIENS DU VIDE, have organised a meeting to take place from 29th May-2nd June 1956 in the Lecture Hall of the Conservatoire National de Arts et Métiers, 292, rue Saint-Martin, Paris 3e, on the subject of high frequency tubes. The design, performance, measurement and production of various high frequency tubes and certain aspects of the technology of linear accelerators will be discussed. Further information is available from Congrès International 'Tubes Hyperfréquences', S.F.I.T.V., 44, rue de Rennes, Paris 6e, France.

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FURTHER LECTURES are announced as follows :

January 3rd, 1956, at 10.40 a.m.
Recent Experiments in the Replication of Surfaces of Fibres.
Lecturers : *L. Oster, J. Sikorski and H. J. Woods.*
Held at : Dept. of Textile Industries, University Leeds.
Arranged by : Institute of Physics, Electron Microscopy Group.

January 17th, 1956 at 7.45 p.m.
High Vacuum Techniques.
Lecturer : *L. G. Beckett.*
Held at: Queen Mary Grammar School, Vyne Road, Basingstoke.
Arranged by : Basingstoke Scientific Society.

February 7th, 1956 at 7 p.m.
Discussion on Purification—Vacuum Distillation of Metals.
Held at : Cadena Cafe, Cornmarket Street, Oxford.
Arranged by : Institute of Metals, Oxford Local Section. Junior Members Evening.

February 8th, 1956 at 6 p.m.
Titanium.
Lecturer : *J, Cueilleron.*
Held at : 28, rue Saint-Dominique, Paris 7e, France.
Arranged by : Centre de Perfectionnement Technique.

February 22nd, 1956 at 6 p.m.
Zirconium.
Lecturer : *R. Faivre.*
Held at : 28, rue Saint-Dominique, Paris 7e, France.
Arranged by : Centre de Perfectionnement Technique.

February 22nd, 1956 at 7 p.m.
Mass Spectrometers.
Lecturer : *Member of Metropolitan-Vickers Electrical Co.*
Held at : Board Room, Chester & District Hospital Committee, 5 Kings Buildings, King Street, Chester.

Vol. 4 195

Arranged by : The Society of Instrument Technology, Chester Section.

February 23rd, 1956 at 5.30 p.m.
Modern Trends in Dairy Refrigeration.
Lecturers : *H. Burley and E. J. Perry.*
Held at : Institution of Mechanical Engineers, 1, Birdcage Walk, London, S.W.1.
Arranged by : The Institute of Refrigeration.

February 24th, 1956 at 7 p.m.
Isolysation and Lyophilisation of Vegetable Materials. Their Application in the Food and Pharmaceutical Industry.
Lecturer : *P. Rovesti.*
Held at : Maison de la Chimie, 28, rue Saint-Dominique, Paris, 7e, France.
Arranged by : Société de Chimie Industrielle.

April 19th, 1956 at 7 p.m.
Ion and Getter Pumps.
Lecturer : *R. Jaeckel.*
Held at : Haus der Technik, Essen, Germany.
Arranged by : Haus der Technik, Essen.

Books

The following books on vacuum subjects have been published recently :

Germany

Physik Duenner Schichten (Part II).
Author : *H. Mayer.*
Published by : Wissenschaftliche Verlagsgesellschaft m.b.H., Stuttgart. (1955)
Pp. 393. Price £6 10s. 0d. (approx.).

United Kingdom.

Vacuum Deposition of Thin Films.
Author : *L. Holland.*
Published by : Chapman & Hall Ltd., London. (1956)
Pp. 541. Price £3 10s. 0d.
(To be reviewed)

Personal

DR. O. S. HEAVENS, the author of the recent book on ' Optical Properties of Thin Solid Films ', has been appointed to the University Readership in Experimental Physics at the Royal Holloway College, University of London.

DR. D. GABOR, Mullard Reader in Electronics at the Imperial College of Science and Technology, London, known for his work in electron microscopy and diffraction has been elected to the Fellowship of the Royal Society.

DR. N. KURTI, Lecturer and Demonstrator in Physics at the University of Oxford, known for his contributions to low temperature research, has been elected to the Fellowship of the Royal Society.

DR. M. PIRANI, who has taken up residence again in Berlin, has recently celebrated his 75th Birthday.

Technical Points

Anti-Reflection Films. The durability of vacuum deposited anti-reflection films on optical surfaces is related to the hardness of the film-forming materials. Figures which indicate the relative hardness of these materials are listed below.

Hardness Scale of Low Refractive Index Materials Used in Anti-Reflection Coatings

Material		*Mean Refractive Index**	*Hardness Number on the Mohs Scale†*
Sodium fluoride	NaF	1.326	3
Cryolite	$3.NaF{\cdot}AlF_3$	1.339	3
Chiolite	$5.NaF{\cdot}3.AlF_3$	1.345	3
Lithium fluoride	LiF	1.392	3
Magnesium fluoride	MgF_2	1.380	5
Calcium fluoride	CaF_2	1.434	4

* For the sodium D-line

† This scale for non-metallic minerals is commonly referred to as the Relative Hardness Scale

●

Low Temperature Physics. High vacuum technique is indispensable to cryogenic research (below liquid air temperatures). Conversely, the results as well as the requirements of low temperature work demand the attention of the vacuum technologist. The following table and others to be published subsequently are intended to provide data which may be required for such work. Accurate values of temperatures of these phenomena are not always readily available and this table will be amended in later issues

as information comes to our notice.

Gas		*Normal Boiling Point °K*	*Melting Point °K*
Helium	He^3	3.2*	—
Helium	He^4	4.21	1 °K at 26 Atm.
Hydrogen†	H_2^1	20.39	14.04*
Deuterium	H_2^2	23.6	18.65‡
Tritium	H_2^3	25.1*	
Neon	Ne	27.2	24.48
Nitrogen	N_2	77.34	63.29
Carbon Monoxide	CO	81.2	66.2
Fluorine	F_2	86.2	50.2
Argon	A	87.5	84
Oxygen	O_2	90.2	54.8
Methane	CH_4	111.8	89.2
Krypton	Kr	121.4	104.2

† These figures refer to hydrogen gas of room temperature equilibrium composition 25% para H_2 and 75% ortho H_2.

‡ Z. Elektrochem., 1938.

* From Low Temp. Physics, C. F. Squire. McGraw-Hill, 1953.

All other values are taken from Smithsonian Physical Tables, 1954. Where conversions have been made from °C to °K the ice point has been taken as 273.15°K.

New Processes and Applications

Coating by Vacuum Evaporation. The development of a principally new type of loudspeaker, the balanced push-pull ELECTROSTATIC LOUDSPEAKER, is reported. Certain deficiencies, inherent in the design of the conventional moving-coil loudspeaker causing energy losses and distortion, are claimed to be overcome by the application of the electrostatic principle. The essential components of the new loudspeaker are the vibrating element which consists of a thin plastic sheet and, situated closely to it, a rigid insulated back plate. One face of both these components is metallised to facilitate electrostatic charging. By applying a polarising voltage the system is kept in electrical equilibrium, until superimposed signal voltages cause the moveable element to vibrate at signal frequency.

It would appear that the task of metallising the two components mentioned constitutes a new potential application of the vacuum evaporation process.

Statistical and Economical

Rare Metals. Zirconium requires vacuum equipment for its production. The hafnium-free zirconium is of particular importance to the reactor designer because of its low thermal-neutron cross section and excellent corrosion resistance at high temperatures. For this reason the bulk of the zirconium produced is hafnium-free.

The major producer country of hafnium-free zirconium is the United States where the Atomic Energy Commission requirements called for 175,000 lbs. in 1951, 900,000 lbs. in 1955 and 1,200,000 lbs. in 1956.

In Great Britain the total production of both hafnium-free and hafnium-containing zirconium to date is 22,500 lbs. per annum.

No actual production figures could be traced for other countries but it is known that some work is carried out in France and Germany. Also, it is reported that Japan has recently offered to supply both the commercial and reactor grades.

Industrial

Germany. E. LEYBOLD - HOCHVAKUM - ANLAGEN G.m.b.H., have raised their capital to 1 million Deutsche Mark. The increase in capital was necessitated by the growth of the turnover of the company. Additional capital was made available in part by Messrs. DEGUSSA (Deutsche Gold-und-Silber-Scheideanstalt A.G.) Frankfurt, who have joined the company of stockholders on that occasion.

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United States and Germany. NATIONAL RESEARCH CORPORATION has entered into an agreement with DEGUSSA (Deutsche Gold-und-Silber-Scheideanstalt A.G., Frankfurt am Main) Germany, for an exchange of information relating to high vacuum furnaces. DEGUSSA has been operating a division concerned with the manufacture of vacuum furnaces for some time.

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United Kingdom. In order to meet the growing demand for complete metallurgical installations, EDWARDS HIGH VACUUM LTD., and EFCO LTD., (Electric Furnace Co. Ltd.) have agreed to pool their long standing experience in vacuum engineering on one hand and electric furnace manufacture on the other, and have decided to form a joint company EFCO-EDWARDS VACUUM METALLURGY LTD.

VACUUM
CLASSIFIED ABSTRACTS

Vacuum Research, Apparatus and Instruments

The publishers endeavour to make the compilation of abstracts published in this section as comprehensive as possible, but cannot undertake to trace and quote all relevant information published on these subjects.

All abstracts are formulated strictly in accordance with the contents of the original publication, but the publishers are not responsible for such mistakes as may have been made in abstracting. Statements or opinions expressed in the publications quoted do not necessarily represent the experience and/or opinions of the publishers.

Arrangement of Subjects

All abstracts published in this section are allocated to one or more of the subjects shown in the following list:

(I) General Science and Engineering

10 — VACUUM ENGINEERING — GENERAL
11 —
12 —
13 — ELECTRICAL SCIENCES
14 — MECHANICAL SCIENCES
15 — PHYSICS
16 — CHEMISTRY
17 — METALLURGY
18 —
19 — SCIENCES (other than the above)

(II) Vacuum Apparatus and Auxiliaries

20 — PUMPS
21 — GAUGES
22 — MEASURING PLANT — VACUUM
23 —
24 —
25 —
26 — MATERIALS
27 —
28 — APPARATUS COMPONENTS
29 — MISCELLANEOUS APPARATUS

(III) Vacuum Processing Techniques

30 — EVAPORATION
31 — CATHODIC SPUTTERING
32 —
33 — IMPREGNATION
34 — FUMIGATION
35 —
36 — DRYING
37 — METALLURGICAL PROCESSES
38 — DISTILLATION
39 — MISCELLANEOUS PROCESSES

(IV) Special Subsidiary Subjects

40 — GASES & VAPOURS
41 — FREEZE DRYING
42 —
43 —
44 —
45 — ELECTRONICS
46 —
47 — GLASS PROCESSING
48 —
49 —

Indices

Three indices are appended to each issue of abstracts: (1) A Cumulative Subject Index in front of the text, (2) a Cumulative Author Index at the back of the text and (3) a List of Periodical Publications quoted. Index (1) and (2) will show all abstracted information published up-to-date in the current volume of the journal.

Filing of Abstracts

All pages containing the text of abstracts are printed on one side only and are detachable ready for filing in special folders. Each main group of subjects (I, II, III, IV) starts on a separate page in each issue and all abstracts in any one group are numbered consecutively throughout all issues of a volume. To distinguish the current numbers of abstracts belonging to one of the four groups from those allocated to other groups, the group number is shown as a suffix to the current number of the abstract, *e.g.*, 20/I as distinct from 20/II, etc.

Translations

Translations of some of the foreign publications quoted can be supplied.

Vol.
4
1954

Cumulative Subject Index, Vol. IV (1954)

For information published previously under these or other headings see October 1951, October 1952, and October 1953 issues respectively.

Subject — Abstract No.

I — General Science and Engineering — I

10 — VACUUM ENGINEERING (GENERAL) — 10

Deaeration ... 10/I;
Distillation ... 7/I;
Freeze Drying ... 16/I;
Gauges ... 3/I;
Gettering ... 2/I;
Leak Detection ... 4/I; 5/I;
Metallisation ... 11/I; 12/I; 13/I;
Metallurgy ... 14/I;
Miscellaneous Applications ... 9/I; 15/I;
Pumps ... 1/I; 6/I;
Vacuum Drying ... 8/I; 9/I;
Vacuum Systems (Design of—) ... 1/I

13 — ELECTRICAL SCIENCES — 13

Carbon Brushes ... 21/I;
Circuitry ... 23/I;
Discharges ... 17/I; 18/I; 19/I; 20/I;
Filaments ... 24/I; 25/I;
Micro-Potentiometer ... 22/I;
Resistors ... 22/I;

14 — MECHANICAL SCIENCES — 14

Design of Vacuum Vessels ... 26/I;

15 — PHYSICS — 15

Boiling Points (Determination of—) ... 38/I;
Cosmic Rays ... 35/I;
Evaporation—General ... 33/I; 39/I;
Interferometry—General ... 28/I; 29/I;
Low Temperature ... 36/I; 37/I;
Magnetism ... 32/I;
Optical—General ... 27/I; 28/I; 29/I;
Photo-Conductivity ... 30/I; 31/I;
Spectroscopy (Infra-Red—) ... 27/I;
Ultra-Violet (Vacuum—) ... 34/I;
Upper Atmosphere (Research of the—) ... 34/I;

16 — CHEMISTRY — 16

Blood and Blood Plasma ... 42/I;
Catalytical ... 44/I; 45/I;
Electrolysis ... 48/I;
Gas Impurities Separable by Condensation (Determination of—) ... 47/I;
Gas Solubility in Mineral Oils ... 46/I;
Hydrocarbons ... 46/I;
Moisture Determination ... 49/I;
Photographic Films ... 43/I;
Surgical and Clinical ... 40/I; 41/I;

Cumulative Subject Index, Vol. IV (1954)

Contd.

For information published previously under these or other headings see October 1951, October 1952, and October 1953 issues respectively.

Subject	Abstract No.

17 — METALLURGY — 17

Calcium	51/I;
Developments	52/I; 53/I; 54/I;
Dezincing of Desilverised Lead Bullion	58/I;
Gas Content of Metals (Determination of the—)	60/I;
Iron and Steel	59/I;
Nickel	57/I;
Platinum	56/I;
Powder Metallurgy	57/I;
Structure of Metals	59/I;
Titanium	50/I;
Zinc	55/I; 58/I;

19 — SCIENCES (other than the above) — 19

Photography	62/I;
Watch Performance at Reduced Pressures	61/I;

II — Vacuum Apparatus and Auxiliaries — II

20 — PUMPS — 20

Developments	1/II;
Diffusion Pump (Oil—)	4/II; 5/II;
Glass Pump	4/II;
Jet Pumps	3/II;
Manifolds	6/II;
Pumping Units (Special Designs of—)	2/II;

21 — GAUGES — 21

Alpha-Particle Ionisation Gauge	8/II;
Developments	7/II;
Diaphragm Manometer	9/II;
Ionisation Gauge	11/II;
Pirani-Type Gauge	10/II;

22 — MEASURING PLANT (VACUUM) — 22

Electron Diffraction	16/II; 17/II; 18/II;
Electron Microscope	19/II;
Mass Spectrometer	12/II; 13/II; 14/II;
Specimen Preparation	19/II; 20/II;
X-Ray Apparatus	15/II;

26 — MATERIALS — 26

Carbon and Carbon Products	22/II; 24/II;
Glycerol	25/II;
Plastics	23/II;
Properties of Materials (Vacuum—)	21/II;
Refractories	21/II;
Teflon	22/II;

Vol.
4
1954

Cumulative Subject Index, Vol. IV (1954)
Contd.

For information published previously under these or other headings see October 1951, October 1952, and October 1953 issues respectively.

Subject	Abstract No.

28 — APPARATUS COMPONENTS — 28

Joints	31/II;
Locks	28/II;
Seals (Other than Shaft—)	30/II;
Seals (Shaft—)	29/II;
Traps	27/II;
Valves (Mechanical—)	26/II;

29 — MISCELLANEOUS APPARATUS — 29

Leak Detector	32/II; 33/II; 34/II;
Liquefier (Gas—)	35/II;

III — Vacuum Processing Techniques — III

30 — EVAPORATION — 30

Bismuth	11/III;
Crucible Techniques	6/III;
Graticules (Manufacture of—)	16/III;
Metals (Deposition of Common—)	1/III; 2/III;
Multiple Layer Films	13/III;
Nickel Films	9/III; 10/III;
Photo-Sensitive Layers	17/III; 18/III;
Physics of Evaporation	6/III; 7/III;
Plastics (Deposition of Metals on—)	5/III;
Properties of Films (Magnetic—)	9/III;
Replica Techniques (Various—)	12/III;
Resistors (Manufacture of—)	15/III;
Sorption of Gases by Films	8/III; 10/III;
Structure of Films	7/III; 11/III;
Thin Films—General	2/III; 3/III; 4/III; 5/III;
Thin Films—Miscellaneous	19/III;
Wires (The coating of—)	14/III;

31 — CATHODIC SPUTTERING — 31

Photo-Sensitive Layers	20/III;
Physics of Sputtering	21/III;

36 — DRYING — 36

Concentration of Liquids (Methods)	22/III; 27/III; 28/III;
Evaporators	22/III; 28/III;
Ovens	23/III;
Puff-Drying	25/III; 26/III;
Refrigerating Plant	24/III;
Theory and Design	23/III;

37 — METALLURGICAL PROCESSES — 37

Alloying Techniques	34/III; 38/III;
Distillation of Metals	32/III; 33/III; 35/III; 36/III;
Evaporation—General	38/III;
Furnaces and Ovens	30/III; 31/III; 34/III;
Fusion Methods (Vacuum—)	37/III;
Melting and Casting	29/III; 30/III; 34/III;

Cumulative Subject Index, Vol. IV (1954)

Contd.

For information published previously under these or other headings see October 1951, October 1952, and October 1953 issues respectively.

Subject	Abstract No.

38 — DISTILLATION — 38

Molecular	40/III; 41/III; 42/III;
Torpidity	39/III;

39 — MISCELLANEOUS PROCESSES — 39

Cooling (Vacuum—)	45/III;
Degassing of Liquids	52/III;
Food Packaging	50/III;
Food Processing	45/III; 46/III; 47/III; 48/III; 49/III;
Geological Age Determinations	53/III;
Rust Prevention	51/III;
Sugar (Processing of—)	44/III;
Testing (Vacuum Methods of—)	43/III;
Vanadium Target (Preparation of—) ...	54/III;

IV — Special Subsidiary Subjects — IV

40 — GASES AND VAPOURS — 40

Analysis	2/IV; 3/IV;
Filling, Mixing and Blending	2/IV;
Gas-Solid Reactions	4/IV;
Helium	1/IV;
Sorption of Gases	4/IV;

41 — FREEZE DRYING — 41

Apparatus and Methods (Various—) ...	7/IV; 8/IV;
Arteries	9/IV; 10/IV;
Blood	5/IV;
Theory and Design	6/IV;
Tissue	8/IV; 10/IV;

45 — ELECTRONICS — 45

Charged-Particle Accelerators	15/IV;
Field Emission	12/IV; 13/IV; 14/IV;
Ion Source	16/IV;
Vacuum Breakdown Phenomena	12/IV; 13/IV; 14/IV;
Valves (Electronic—)	11/IV; 12/IV;

47 — GLASS PROCESSING — 47

Glass-to-Metal Seals	17/IV;

Vol.
4
195

I — General Science and Engineering — I

Abstract No. and References

10 — VACUUM ENGINEERING (GENERAL) — 10

A Portable High Vacuum System 1/I

United States. A portable high vacuum unit is described which was developed to facilitate demonstrations in connection with lectures, etc. The base plate of the unit measures no more than 12×19 inch. The case is similar in appearance to the carrying case of a 16 mm. cine-projector and houses a glass diffusion pump, a fore pump, a cold trap, a Variac, a ground glass joint, a spark coil and an ionisation gauge. To save space the cold trap is built into the high vacuum side of the pump and can be charged with either liquid air or dry ice. For operation over night a special Dewar-type flask could be used to cool the trap. The Variac facilitates the adjustment of the diffusion pump heater. By building-in small blowers the water-cooled diffusion pump may be replaced by a 2-stage air-cooled pump. The standard unit produces an ultimate pressure of 10^{-6} mm. Hg but by replacing the ground glass joint with a metal Alpert valve and gauge (see Vol. III, Abstract No. 191/I), pressures of the order of 10^{-10} mm. Hg may be obtained. The complete unit weighs 40 lb. It can be operated on a 220 V or 115 V supply.

Article by V. C. DeMaria *Sylvan. Tech.* 6, Oct. 1953 96-97

Sommaire : La description d'une machine transportable, donnant un vide de 10^{-6} mm. Hg, equipée d'une jauge à ionisation, fixée par raccord de verre, est donnée. Son poids est d'environ 20 kg.

High Temperature Materials for Vacuum Service

See Abstract No. : 21/II

Techniques of Measuring and Controlling Vacuum 3/I

United States. The types of instruments available for the measurement and control of pressures below atmospheric are reviewed. There are two groups of designs: The direct measuring gauges actuated by the force exerted by the pressure to be measured and the inferential measuring gauges actuated by some other physical property of the gas to be measured which changes with its pressure. The following instruments belong to the first group : (*a*) The Spiral Gauge. It consists of a flat Bourdon tube wound in the form of a spiral, the end movement of which is transmitted by a single link to the pointer. The range of the gauge is 6.5-15 lb./sq. inch. (*b*) the Spring-and-Bellows Gauge for intermediate (30-10 inch. Hg) and in a special form, for still lower pressures. The instrument for intermediate pressures consists of a bellows situated in a bellows shell. The gas, the pressure of which is to be measured, is admitted to the space between shell and bellows and causes an upward movement of the bellows against a spring. A rod fitted to the bottom of the bellows is linked with the indicator. (*c*) The compound Spring-and-Bellows Gauge. This gauge consists of two bellows. The ends of the bellows facing each other are joined to a common moveable plate whereas the other two ends are rigidly fixed to the housing. One bellows is evacuated and sealed and contains a calibrated spring. The open end of the other admits the gas, the pressure of which is to be measured. The instrument gives absolute pressure readings in the range 0-100 mm. Hg. (*d*) The Inverted Bell Gauge. This gauge is very sensitive but covers only a narrow range. The single bell-type is suitable for the measurement of static pressures and the double-bell type for the measurement of differential pressures as required in air flow measurement, for instance. The bell is inverted and suspended in oil. The pressure of the gas to be measured causes a movement of the bell which is transmitted to a counterweighed balance beam linked with the indicator. The double-bell design has a sensitivity of ±0.0005 inch of water. Scale ranges of 0–2 inch. to 0–5 inch. of water are available. (*e*) The McLeod type vacuum gauge, which covers the pressure range 10^{-6} to 1 mm. Hg. The working principle of the gauge is described. The gauge can be made to record continuously by electrical means. The group of inferential measuring gauges comprises: (*a*) The Pirani (Hot-wire) gauge. Its normal range is 10^{-3}–1 mm. Hg. Details of the working principle are given. (*b*) The Thermocouple Gauge. Four filaments are heated uniformly but independently from four secondary windings of a transformer. Two of these filaments are mounted in the measuring chamber of the gauge and the other two in a reference chamber sealed after evacuation to 10^{-3} mm. Hg. A thermocouple is connected in series with each pair of filaments and the two thermocouples are connected together so that their outputs are opposed. The difference between the two opposing output e.m.f's is a measure of the difference in pressure between the reference and measuring chamber. For a pressure variation of 1 to 500 micron Hg. the e.m.f. changes from 0 to about 19.2 mV. (*c*) The Hot-Cathode Ionisation Gauge. The lower limit of this gauge is below 10^{-4} mm. Hg. Details of its design are discussed. (*d*) The Alphatron Cold Cathode Ionisation Gauge. As in the case of (*c*) the operation of this gauge is based on ionisation phenomena in the residual gas but, instead of electrons, alpha particles emitted by a quantity of radium placed in the measuring chamber of the gauge, serve as the ionising means. The range of the instrument is approximately 10^{-3} mm. Hg–10 mm. Hg.

Article by C. C. Roberts & W. W. O'Hara *Prod. Engng.* 24, Sept. 1953 190-194

Sommaire: Une revue génerale des appareils de mesure et de controle du vide couvrant toutes les pressions depuis le micron jusqu'au millimètre.

A Mass Spectrometer for Leak Detection 4/I

See Abstract No. : 33/II

I — General Science and Engineering — I

Contd.

Abstract No. and References

Experience Gained with Various Leak Detection Methods and Apparatus

5/I

Germany. The most popular method of leak detection today is the probe gas method. A gas, not contained in atmospheric air, is sprayed on to the outside of the walls of the evacuated vessel and on reaching the interior of the vessel its presence is recorded by an instrument which either reacts to that gas only or reacts to the gas differently than to atmospheric air. In all cases the leak detector apparatus records the partial pressure of the probe gas in the vessel and it is erroneous to define the sensitivity of a leak detector by the minimum size of leaks it will trace. The larger the volume of the vessel under test, the longer it takes to obtain equilibrium conditions. In order to reduce the time spent in the operation large capacity diffusion pumps should be used and the leak detecting instrument should be connected to the backing line. Assuming that the volume of that line equals 0 the partial pressure of the probe gas present in the backing line is

$$p = \frac{L}{S_v}\left(1 - e^{-\frac{S_d}{V} \cdot t}\right)$$

where L is the size of the leak in mm. Hg litre/sec., S_v is the pumping speed of the fore pump, S_d the pumping speed of the diffusion pump, V the volume of the vessel under test and t the time employed for spraying the probe gas on to the wall of the vessel. In this arrangement S_d/V is nearly 1, thus the times required to obtain proper equilibrium conditions are of the order of approximately 1 second. Conventional vacuum gauges, such as ionisation and hot-wire gauges can be employed for the detection of leaks as long as hydrogen is used as the probe gas. Their sensitivity in that capacity is equivalent to that of their normal functions and this is satisfactory for many cases, for instance, in the case of continuously pumped vessels. But, if the vessel is not continuously pumped or, if the volume of the system is very large, apparatus of higher sensitivity are required such as the mass spectrometer leak detector. With helium as the probe gas this instrument registers 10^{-9} to 10^{-10} mm. Hg probe gas partial pressure. The apparatus can be used with other gases but only at the expense of its sensitivity. Another special leak detector is the palladium barrier (ionisation) gauge. The palladium disc separating the gauge from the system is kept at a high temperature and in that state permits the penetration of hydrogen only to the gauge. But hot palladium tends to absorb oxygen. As a result, oxygen and hydrogen combines and the readings of the instrument are safe only if the oxygen partial pressure is below 10^{-5} mm. Hg. If working properly the sensitivity of the instrument is 10^{-6} to 10^{-7} mm. Hg. A fairly new instrument is the American halogen-platinum leak detector which is sensitive to a test gas partial pressure of 10^{-8} mm. Hg. Here, the probe gas is a refrigerant of the formula CF_2Cl_2 (Freon). In operation the refrigerant gas contacts the hot platinum and causes the emission of positive ions. This effect is independent of pressure and the instrument can be used to trace leaks at atmospheric pressure by employing a special technique. A table, partly reproduced below, gives a comparison of the smallest leaks traced in a typical vacuum plant by employing the various methods described in the article. The spraying time was 1 second in all cases.

Plant	*Hot Wire Gauge with Hydrogen mm. Hg l./sec.*	*Ionisation Gauge with Hydrogen mm. Hg l./sec.*	*Halogen-Platinum Leak Detector with Freon (CF_2Cl_2) mm. Hg l./sec*	*Leak Detector Connected to*
Evaporation Plant V =200 l. S_v = 15 l./sec. S_d =400 l./sec.	4×10^{-2}		4×10^{-7}	Mechanical Pump
	2×10^{-1}	2×10^{-4}	2×10^{-6}	High Vacuum Diffusion Pump
	1.5×10^{-2}		1.5×10^{-7}	Backing Side of the Diffusion Pump

Sommaire : Toutes les méthodes existantes de détection des fuites sont passées en revue, et leurs qualités sont comparées.

Article by K. Ziock *Glas & Hochvakuum Tech.* **2**, Dec. 1953 292-294

Pick a Vacuum System for Your Job

6/I

United States. General advice is given to the chemical engineer on the choice of a suitable vacuum pump for processes to be carried out at sub-atmospheric pressures. The pumping of non-condensable gases and vapours only is considered in this article. The main factors governing the choice are:—The processing pressure, the vapour volume to be pumped, the pumping-down time allowed and operating hours per year. Reciprocating piston-type pumps cannot be used for ultimate pressures below 10 mm. Hg since the necessary clearance between the piston and the cylinder head permits re-expansion but they can handle vapour volumes of 10,000 cu. ft./min. plus. The oil-sealed mechanical pump has a piston which revolves eccentrically in a cylindrical housing. The piston is fitted with a slide separating the inlet and exhaust port of the chamber. The pump can produce an ultimate vacuum of approximately 10^{-3} mm. Hg and handle vapour volumes up to 1,000 cu. ft./min. The ejector pumps entrain the vapours to be exhausted in a high velocity stream of the pump fuel which is preferably steam. The steam ejector can produce an ultimate vacuum in the range between 10^{-2} and 10 mm. Hg and can be designed for capacities up to 100,000 cu. ft./min. But its use is largely governed by the existing supply conditions regarding steam and water. All these three pumps can work directly against atmospheric pressure. A special form of the ejector pump, the diffusion pump, requires low pressure on the

I — General Science and Engineering — I
Contd.

Abstract No. and References

exhaust side, normally produced by a mechanical pump, for its operation. In this pump a ' curtain ' of slow moving oil or mercury vapour, raised in a boiler, entrains the air molecules diffusing into it. The vapour of the pump fluid is condensed on a water-cooled surface and returns to the boiler as liquid. The air liberated on condensation, is carried away by the fore pump. This type of pump is indispensable, where high vacua and capacities exceeding 1,000 cu. ft./min. are required. The larger the diffusion pump, the greater is the size of the mechanical pump required. In order to keep the mechanical pump to a reasonable size, it is often advisable to place an oil ejector pump as a ' booster ' between the diffusion and the mechanical pump. A graph gives an indication of the initial costs per cu. ft./min. for both diffusion and mechanical pumps. Estimating pump-capacities at low pressures, a safety factor of 3 should be used. Finally, vacuum valves are discussed and the designs available for various applications are shown in a table, reproduced below.

Pressure Range	*Up to ½ inch Diameter*	*½ inch to 6 inch Diameter*	*6 inch Diameter and Larger*
Less than 10^{-3}mm. Hg	Glass Stopcock Needle Refrigeration type	Bellows	Disk
Greater than 10^{-3}mm. Hg	Needle Globe Gate Refrigeration type	Globe Rubber diaphragm Bellows	Globe Disk

Sommaire : Un avis général est donné sur le choix de pompes appropriées aux différentes exigences de procédés de fabrication chimique.

Article by L. W. Hull *Chem. Engng.* **60**, Nov. 1953 200-202

Some Aspects of Molecular Distillation 7/I

See Abstract No. : 40/III

Moisture Removal 8/I

United States. The increase in the annual production of citrus concentrate from 200,000 gal. in 1946 to 50,560,000 gal. in 1953 is an indication of the important developments which have taken place in the field of food concentration and drying techniques in recent years. Present day methods carried out at atmospheric and reduced pressures are reviewed. Three new vacuum drying methods are described. (1) The liquid is de-aerated in a vacuum chamber then spread as a film on to a stainless steel belt which passes first around a steam-heated drum and then around a cooled roll. The finished product is removed from the belt by a scraper and has a moisture content of 1–2%. The operational pressure is 5 mm. Hg produced by a steam-ejector system. In the case of drying a 40% coffee concentrate the total time cycle is 27 sec. and 65-70 lb. of soluble coffee are produced per hour in one unit. (2) Represents a modified freeze drying process. The concentrate to be dried is ejected in small pellets into a refrigerant bath (Freon at–40 to–55°F). The frozen pellets are collected from the surface of the refrigerant, passed into the vacuum chamber, cleaned of all surplus refrigerant and fed on to belt conveyors which carry the material through the dryer or sublimator. The heat is supplied by infra-red radiation. The pressure in the chamber is 5–6 mm. Hg. The vapours are removed by condensation. (3) Represents a typical ' Puff-drying ' process (see also Vol. III, Abstract No. 76/III). It follows a discussion of up-to-date concentration methods. 35 years ago, 250 gal. of single strength citrus juice were produced p. man hour. To-day, one operator can control plant of an input of 5,000 gal. p. hour. The most important advances were obtained by introducing low-temperature evaporation methods. Some of the characteristics of these methods are : Operation at reduced pressures, *i.e.* 8–20 mm. Hg, exposure of the material to heat in the form of a ' falling film ' and use of multiple-effect evaporators. Special mention is made of the heat-pump evaporating system. The embodiment of this principle provides for the hot ammonia gas, used in the refrigeration compressors, to supply the heat of evaporation to the unit where the juice is concentrated and the vapours of the refrigerant are condensed. In another method developed in Switzerland for use with excessively foaming materials, the liquid is thrown against the inner surface of the heated evaporator shell by blades rotating at a controlled speed. The clearance between the blades and the cylinder wall is 1/32 inch and the time, the falling liquid film is in contact with the heated surface, is 5 to 7 seconds; this facilitates operation at higher temperatures than normally employed. This system has been used for processing caramel candy, fruit juices, gelatin, coffee, vitamins and antibiotics.

Sommaire : Une revue générale des méthodes modernes de concentration et de séchage dans le traitement industriel des produits alimentaires avec référence spéciale sur la production du jus de fruit.

Article by Anon. *Food Engng.* **25**, Oct. 1953 143-147 287-288

Moisture in Refrigerant Machinery Circuits 9/I

See Abstract No. : 24/III

I — General Science and Engineering — I
Contd.

Abstract No. and References

Rust in Water System Checked by Deaeration

10/I

United States. To protect the steel piping at the U.S. Naval Hospital, St. Albans, N.Y., a vacuum deaerator and 100,000–gallon elevated storage tank have been installed. The steel deaeration tank, 7 ft. in diameter and 20 ft. high, contains multiple layers of wood slats over which the water is discharged from spray nozzles. A steam injector maintains a vacuum of at least 29.1 inch. Hg. The water obtained from wells is saturated with oxygen and contains 15 p.p.m. free carbon dioxide with a temperature as low as 50°F. The desired oxygen and free carbon dioxide contents are 0.3 and 5 p.p.m., respectively. A floating cover of the open-pan type in the elevated tank prevents reabsorption of gases from the atmosphere. Unpleasant tastes and odours due to anaerobic conditions have not resulted.

(*Chemical Abstracts*)

Note by J. L. Staunton *Engng. News Reg.* 151, No. 9, 1953 42-43

Sommaire : Des détails sont donnés sur un appareil de désaeration par le vide, construit dans le but de désaerer l'eau alimentant un hospital, afin de retarder la formation de rouille dans les conduites de distribution.

Metal Finishing by High Vacuum Metallisation

11/I

See Abstract No. : 2/III

Economical Coatings by Vacuum Metallising

12/I

See Abstract No. : 5/III

Method and Equipment for the Deposition of a Metal Layer of Wire or Similarly Shaped Materials

13/I

See Abstract No. : 14/III

Development of Commercial Vacuum Furnaces for Melting Metals and Alloys

14/I

United States. The advantages offered of purifying metals by melting them in vacuum were first established by Arsem in 1906. The initial design of the Arsem furnace was used over a long period for all forms of metallurgical reduction and refining but it did not provide any facilities for casting *in vacuo*. Up to the first World War the furnace was equipped with a Guericke mechanical pump which produced an operational pressure of 0.5 mm. Hg. At a later stage the graphite resistor heating element employed in the early design was replaced by a high-frequency induction coil. The modified furnace was fitted with a plugged outlet at the base of the crucible leading to an ingot mould (*Metal Prog.*, Sept. 1947). After the first World War high frequency heating in vacuum furnaces was adopted generally in the United States, together with the newly developed high vacuum pumps, whereas Germany built furnaces fitted with low-frequency induction heating up to a capacity of 8,000 lb. for some time. The three basic types of modern vacuum furnaces are shown in diagrams. They differ mainly in the method of casting. In one case the furnace as a whole is tilted. In the second, the furnace chamber is stationary and the crucible only is tilted and in the third, a bottom-tapped crucible is employed. As a result of improved vacuum pumps performance the size of the vacuum furnace could be increased. During the second World War furnaces were constructed in the United States for charges up to 300 lb. heated by 10,000 c.p.s. 200 V induction coils, fitted for bottom pouring into water-cooled copper moulds. Further study of the tilt-pouring technique led to the design of furnaces for semi-continuous operation. A unit of this type (*U.S. Pat.* 2,625,719) is described and shown in a sketch. It contains certain devices, such as locks, which permit charging and discharging without breaking the vacuum in the furnace chamber and interrupting the melting process. The operational pressure in the unit is 1 micron Hg. More recent designs of such furnaces can produce 1,000 lb. ingots of copper alloys and 600 lb. ingots of steel.

Article by J. H. Moore *Metal Prog.* 64, Sept. 1953 161-164

Sommaire : Un resumé de l'histoire de développement des fours à vide est donné.

The Influence of Barometric Pressure on Watch and Chronometer Rates

15/I

United Kingdom. The barometric pressure may change by an amount of up to 10 mm. Hg per day. Unless a watch is truly isochronous, pressure changes affect the rate of the watch. The pressure coefficient is known to vary from 0.007 to 0.02 sec./day per mm. Hg and thus the effects of pressure changes on performance may be equivalent to effects from other causes such as temperature changes. In order to obtain experience on the question, whether pressure allowances should be made in precision tests, the National Physical Laboratory conducted practical experiments on a Kullberg marine chronometer and on a Ditisheim watch at various sub-atmospheric pressures. The time pieces were placed in a chamber, fitted with an observation window, which could be exhausted. The end positions of the balance, determining the arc, were observed by means of a microscope with a rotating head. The rate was measured by picking up the ticks through a microphone, amplifying the signals and comparing them with those obtained from an N.P.L. standard quartz oscillator clock. An oscillating balance wheel can be affected by pressure changes in two ways. As the pressure rises, the amount of air carried around by the wheel grows, the movement of inertia increases and, corresponding to it, the period of oscillation. On the other hand, the amplitude of the swing, *i.e.* the arc, will be reduced and this will be accompanied by a change of period. In the experiments the changes of rate caused by (artificially produced) changes of arc at atmospheric pressure were determined first. Then the rates were measured and plotted for various sub-atmospheric pressures. Finally, taking into account the changes of rate due to changes

Vol. 4 1954

I — General Science and Engineering — I
Contd.

Abstract No. and References

of arc, rate/pressure curves at constant arc were derived. The experiments prove that the time pieces gain steadily as the pressure is reduced. In the case of the chronometer the pressure coefficients were found to be 0.013 sec./day/mm. Hg in the 100-200 mm. Hg pressure range and 0.009 sec./day/mm. Hg in the pressure range of 600-700 mm. Hg. The results for the watch were similar: The pressure coefficients of the watch were found to be 0.021 sec./day/mm. Hg in the low pressure range and 0.012 sec./day/mm. Hg in the high pressure range. The authors came to the conclusion that the pressure relationships are too complicated to introduce pressure allowances in precision tests. All tests should be carried out in a chamber isolating the instrument from the free atmosphere.

Sommaire : Les pendules et autres appareils de mesure du temps sont connues comme étant perturbés par les variations de pression atmosphérique. Des expériences sont rapportées, ayant été faites dans le but de savoir s'il est nécessaire d'en tenir compte durant les tests.

Article by N. W. B. Stone K. W. T. Elliott & P. H. Bigg *Brit. J. Appl. Phys.* 4, Dec. 1953 379-382

Spare Parts for Humans — 16/I

United Kingdom. The first operation during which bone fragments taken from one patient were successfully transplanted into the body of another patient was carried out as early as 1878 but further use of this operative technique was greatly hindered by three factors: (*a*) The danger of infection, (*b*) the danger of blood clotting and (*c*) the lack of means whereby tissues could be stored successfully for a reasonable period. (*a*) Was overcome after the advent of antibiotics, (*b*) by the development of suitable drugs and (*c*) by employing the freeze drying technique, which was eventually responsible for the establishment of the tissue bank system. Bone banks are now in existence where bone made available through amputations or death is freeze-dried and stored and from which it can be shipped to any hospital in the world, in need of supplies. Transplants using freeze-dried bone have been successful in 85% of the cases in Britain. Preserved bone will not grow in the body of the host but it provides a ' trellis ' along which new bone grows and gradually absorbs the transplanted bone. Artery banks are among the most vital of all tissue banks. Arteries to be stored must be taken from the donor not later than 6 hours after death. The staff of the artery banks is on call day and night for the purpose. Freeze-dried arteries can be stored for a period up to one year whereas previous methods facilitated a maximum storage period of 6 weeks only. As with bones, arteries after transplantation are gradually absorbed by the body without growing themselves. The latest developments are skin banks for the supply of skin required for grafts in serious cases of burns, for instance. Experiments are in progress to freeze-dry skin in order to lengthen the storage period. Finally, cartilage banks, used mainly by surgeons repairing noses, ears and foreheads are mentioned. The surgeons found that bovine cartilage is quite as satisfactory as human cartilage and Armour Laboratories, Chicago, have started to market regularly bovine cartilage for transplantation in humans.

Sommaire : Les emplois et qualités des systèmes variés de banque des tissues en existance dans le domaine médical, sont décrits.

Article by J. D. Ratcliff *Reader's Dig.* 63, Nov. 1953 52-54

13 — ELECTRICAL SCIENCES — 13

Discharge Mechanisms in Cold Cathode Valves — 17/I

See Abstract No. : 12/IV

On the Mechanism of the Initiation of an Electrical Gaseous Discharge Under the Action of a Transverse Magnetic Field in the Pressure Range 10^{-1} to 10^{-8} mm. Hg — 18/I

Austria. A theory is evolved in which the electrical field is inhomogeneous (cylindrical electrodes) and the mean free path is large compared with the ' rolling ' diameter of the electron's cycloidal path. The ignition characteristic (initial potential as a function of the magnetic induction multiplied by the electrode gap at a given pressure) is calculated and compared with the measurements on argon in the pressure range 10^{-1} to 10^{-8} mm. Hg. At all points there was agreement between theory and experiment.

(*Nuclear Science Abstracts*)

Sommaire : Une étude théorique des caractéristiques d'allumage d'une décharge dans un champ magnetique transversal sous des pressions très basses.

Article by R. Haefer *Acta Phys. Austriaca* 7, Oct. 1953 251-277

The Field-Emission Initiated Vacuum Arc. I. Experiments on Arc Initiation — 19/I

See Abstract No. : 13/IV

The Field-Emission Initiated Vacuum Arc. II. The Resistivity-Heated Emitter — 20/I

See Abstract No. : 14/IV

I — General Science and Engineering — I
Contd.

Abstract No. and References

Wear of Carbon Brushes at High Altitudes

21/I

United Kingdom. Brushes on generators used in aircraft flying at moderate heights wear at a rate of 0.0001 to 0.001 inch p. hour. The surface of the brush shows an orientation of the graphite crystallites consistent with the rubbing action. However, when the generators are flown at about 30,000 ft. the rate of wear rises up to a value of 0.02 inch p. min. *i.e.* by a factor of 1,000 and the brush surface is rough and disorientated. Also, the commutator surface has the appearance of raw copper covered with a layer of graphite. This phenomenon was found to be independent of electrical conditions and therefore could only represent the effect of mechanical action. At the same time it proves that Bragg's conception of the properties of graphite as a lubricant do not apply and that atmospheric conditions play an essential part. Bowden and Young have shown that the friction between two surfaces increases rapidly when the surfaces have been freed of all contamination by heating *in vacuo*. It is now recognised that in normal atmospheric conditions cohesion between brush and commutator is prevented by the presence of a film of adsorbed carbon on the commutator. Savage claimed that this film is absent at high altitudes and supported this view by experiments (see *Gen. Electr. Rev.*, **48**, 1945, 13) producing high friction and rapid wear during operations in an evacuated chamber, freezing out all moisture with a liquid air trap. The rate of wear rose sharply when the water vapour pressure reached the critical value of about 3 mm. Hg or less. The critical values for other potential lubricants in the form of vapours or gases were found to vary between about 5 mm. Hg for ammonia and acetone, and about 600 mm. Hg for hydrogen and nitrogen. Based on these findings of previous workers the Royal Aircraft Establishment investigated the conditions methodically by studying the performance of the brushes of (blast cooled) generators of the aircraft type at pressures down to 33 mm. Hg (=70,000 ft. altitude), varying temperatures down to –80°C and varying current densities, spring pressures and speeds. The test equipment consisted of three bell-jar vacuum chambers housing the slip ring—brush sets under test. The motors driving the sets were mounted outside the chamber. Air, refrigerated to the desired degree, was driven through each chamber by a vacuum pump. Heating elements were provided in the brush boxes and the brush temperature was measured by embedded thermocouples. In an experiment, running electrographitic brushes on copper at 130°C, it was confirmed that the rate of wear falls rapidly as soon as the water vapour pressure rises beyond a well defined value and if the water vapour pressure is constant the rate of wear rises as the pressure is reduced. Rapid wear can occur at altitudes above 10,000 feet but it has rarely been experienced below 20,000 ft. A set put into operation at high altitude shows excessive wear until the pressure is raised to a certain value, above which it will fall to a negligible amount. This pressure level is termed ' critical altitude '. If a set has been in operation for some time before it reaches the critical altitude an adsorbing film will have formed and excessive wear will set in only at greater heights. The actual values of critical altitude and pressure vary considerably with temperature. In order to improve lubrication the brushes were treated with various adjuvant materials. Tests showed that oils and other organic substances were effective only at low temperatures. But the use of metallic halides shows a marked improvement. For instance, barium fluoride treated brushes will stand up to –65°C at 50,000 ft. if they have been run-in for 12 hours at low altitudes and at a temperature of 170°C. It appears that the protection obtained by this treatment is due to the formation of a film on the commutator surface by a chemical process.

Sommaire : Des balais de carbone de générateurs placés dans des avions volant à grande altitude presentent une usure excessive. Des détails de recherches faites sous les mêmes conditions sont donnés et des remèdes possible sont discutés.

Paper by R. F. Sims *Engineer* 196, 18.12.1953 823-824

Fabrication of Radio-Frequency Micropotentiometer Resistance Elements

22/I

United Kingdom. The radio-frequency micropotentiometer has been developed to provide voltage standards in the micro-range. Its accuracy is $\pm 1\%$. The instrument has a low-output impedance ranging from 1 to 10^3 milliohms. The low-resistance elements required are thin film resistors in the form of discs. These resistors have to have a good stability over long periods and a satisfactory flat frequency characteristic up to at least 300 Mc. Films applied by painting at low-temperatures and subsequently baked were unsatisfactory. The performance of silver films applied by chemical reduction has not yet been established. The present report describes three different methods:—(*a*) Clamping of carbon discs, (*b*) high temperature firing, (*c*) evaporation and plating. It is important that the film is even and that good contact is made between the electrodes and the film as otherwise the r.f. impedance of the resistor element is frequency-sensitive. All these methods yield films of satisfactory stability and the current-carrying capacity of the films is five times that normally used in the instrument (100 mA). The mean-temperature coefficient of resistance varies between 3×10^{-1} (% in resistance p. °C) in the case of evaporated and plated silver films and 8×10^{-3} in the case of carbon sheet, 25 ohm. p. square. Varying the thickness of the films, a resistance range of about 10 to 1 may be obtained. In method (*a*) discs are cut from a Bakelite sheet carrying a thin carbon film, stacked, clamped between flat metal plates, drilled in the centre and turned on a lathe. Then all discs are coated with a conductive lacquer (except for a small area in the centre) in order to make contact with the electrodes when the discs are clamped within a brass shell. This method is not satisfactory for low-value resistance units. Where elements of less than 1 ohm are required method (*b*) can be used. Here a short glass or ceramic tube is painted by hand (except for one end) while rotating in a motor-driven jig. The film is fired at about 1,400°F and then burnished and cleaned. The finished element is soldered to a cup-shaped electrode at one end and to a pin-shaped electrode at the other. Resistor elements of approximately one milliohm are produced by method (*c*), *i.e.* by evaporating and subsequently electroplating silver on to coaxial electrodes and the dielectric separating the electrodes from each other. Kovar-glass sealed terminals can be used in this method. The Kovar surface is freed from tin and

Vol. 4 1954

I — General Science and Engineering — I
Contd.

Abstract No. and References

electro-plated with silver first. Then, the resistive silver film is deposited by vacuum evaporation and this is followed by plating a silver top layer of 2.5 μ thickness. Finally a protective lacquer is applied. The production of 1–5 ohm elements by method (*b*) appears to be simpler than the production of 1–10 ohm resistances by method (*a*).

Sommaire : Trois méthodes, dont une application de procédé d'évaporation sous vide, pour la production de résistances sous forme de couches minces du type employé dans les micropotentiometers r.f. sont décrites.

Article by L. F. Behrent *J. Res. Nat. Bur. Stand* 51, July, 1953 1-9

A Few Remarks on the Electronic Circuit of a Leak Detector 23/I

France. The electronic circuits of a leak detector constructed at the Commissariat à l'Energie Atomique (see Abstract No. 33/II) are described. The power supplies for the ion source are of a conventional type. No high stability is required. A multivibrator supplies a saw-tooth voltage at 10 c.p.s., which is superimposed on the ion accelerating potential. The ion current is first amplified by a high gain a.c. amplifier employing a high degree of negative feed-back. An additional a.c. amplifier for further amplification of signal is used. The spectrogram can be observed directly on the screen of a cathode-ray tube by connecting the horizontal deflection plates with a sweep generator and the vertical plates with the output of the amplifier. The main improvements compared with other circuits concern the design of the input stage of the pre-amplifier. In order to reduce stray capacity between the ends of the input resistor and earth and the body of the input resistor and earth, the resistor has been placed between two parallel metal plates. Holes in the centres of the plates allow the ends of the resistor to pass through. The plates are suitably connected with the earth and the output of the pre-amplifier respectively, in order to form an electrostatic compensating field around the resistor. The relative position of all three plates and the resistor, can be adjusted to obtain better compensation. In this way a lower time constant is obtained, *i.e.* a faster response is obtainable and the results are more reproducable. The electrometer tube is mounted on an elastically suspended heavy metal plate to protect it against vibration. It is also protected from light to reduce the photo-electric emission of the grid. The pre-amplifier has a gain of 10,000, and its pass band is limited to below 80 c.p.s., to reduce internal tube noise. The signal-to-noise ratio was thus improved by a factor of many hundreds compared with the usual designs of amplifiers employing the negative feed-back principle. Thermal drifts have been much reduced and it is therefore not necessary to wait for the instrument to stabilise after switching on. It has been possible to observe ion current peaks of the order of 5×10^{-14}A with any electrometer tube of the type 959.

Sommaire : Caractéristiques essentielles des circuits electroniques associés au detecteur de fuites construit au CEA (Vois Résumé No. 33/II).

Article by J. Lecomte J. Taieb & J. Thébault *Le Vide* 8, Nov. 1953 1422-1427

Repairing Ionisation Gauge Tubes 24/I

See Abstract No. : 11/II

Improvements i.o.r.t. Apparatus for Measuring Gas Pressure 25/I

See Abstract No. : 10/II

14 — MECHANICAL SCIENCES — 14

A Metal High Vacuum Manifold 26/I

United States. A vacuum manifold is described which can be fabricated in less than 3 hours from metal components. It essentially consists of 6 Veeco packless angle valves which are joined to copper pipes by T-joints and are fitted with brass ball joint male leads. All joints are silver-soldered. The type of valve used has a 1-inch bore and is machined from 2-inch. square brass stock. Its length is $3\frac{13}{16}$ inch. The valve stem is sealed vacuum tight by means of a bellows. The valve disc makes line contact with the valve seat. Synthetic rubber gaskets or Teflon gaskets are used for the pipe connections to the vacuum systems. Illustrations of the valve and the manifold assembly are given. In order to test its vacuum characteristics, the manifold was connected to a glass vacuum system which consisted of a three-stage oil diffusion pump with a speed of 8 l./sec. at a pressure of 1×10^{-2} mm. Hg, coupled with a mechanical fore pump, and a cold-trap and ionisation gauge in the pumping line. Two series of tests were carried out. The first was conducted after exposing the manifold to atmosphere for 24 hours. The second, after exposing the manifold to atmosphere for 12 minutes subsequent to a previous pumping period. In both experiments the manifold was pumped for 30 minutes. The pressure obtained in the first experiment was 9.6×10^{-5} mm. Hg and in the second 4.6×10^{-5} mm. Hg. The ultimate pressure obtainable was reached after 36 hours and was determined at 4.6×10^{-6} mm. Hg. In another experiment the manifold was pumped for 8 hours and then sealed off. After 2 hours in the sealed-off state the pressure had risen from 5×10^{-5} mm. Hg to 8.2×10^{-4} mm. Hg. The manifold described should be of greater use than glass manifolds in all laboratory work requiring vacuum apparatus except in processes where gas–phase reactions may cause deposits on the metal surfaces of the new design.

Sommaire : Description est donnée d'un manifold en métal pour vide poussé dont les caractéristiques sont telles qu'il pourrait remplacer les manifolds de verre dans les travaux normaux de laboratoire.

Report by E. Paige *AEC Report No. MLM-810* Feb. 1953

I — General Science and Engineering — I
Contd.

Abstract No. and References

15 — PHYSICS — 15

The Production of Diffraction Gratings 27/I

United Kingdom. Diffraction gratings are important aids in spectroscopy and consist of flat or spherical mirrors ruled with grooves of a given cross-section at a density of 1–30 thousand parallel grooves per inch. The gratings replace prisms in spectroscopes and deflect the incident light energy of a complex radiation, singling out individual wavelengths and thus giving information on the nature of its components, by analysing complex radiation. In the past these gratings were manufactured by a method, first described by H. A. Rowland in 1870, which was based on the use of a device working generally on the principle of a dividing engine. The work-holder with the plate to be inscribed is moved in steps by a lead screw, operated by a rachet wheel. The cutting tool, a diamond, inscribes the plate in a reciprocating action while the work is at rest. Due to the tolerances inherent in this device, the accuracy required in inscribing gratings, *i.e.* 10^{-6} inch, cannot be maintained and ghost lines are produced in the spectrum. In the period immediately following the last war the demand for optical gratings rose appreciably and this led Sir Thomas Merton (in 1948) to propose a new method, *i.e.* to cut one continuous fine screw thread on a cylinder and to produce a flat grating from this helical grating by a special copying method. After cutting the helix a plastic solution (polystyrene) is placed around the helix. After hardening, this ' skin ' is cut open and placed on a flat surface of moist gelatine which during the drying process penetrates the pellicle and produces a permanent copy ' in the flat '. In this condition the grating can be permanently used as a transmission grating or alternatively, is coated with an aluminium deposit by vacuum evaporation in order to serve as a reflecting grating. The early helices produced by this method had up to 15,000 turns per inch but still showed periodic errors which were due to the vibrations caused by the inevitable tolerances in the machine. In order to overcome this the ' Merton nut ' was introduced. In the final Merton method a helix to the required dimensions is cut along one half of the length of a polished metal bar. This part of the bar is then fitted with the Merton nut and revolved but the nut is prevented from rotating with the bar. The grooves of the threads of the nut are fitted with a resilient material. This facilitates pressing the nut firmly into the grooves of the helix assuring a perfect fit all the time. As a result the periodic errors of the primary screw are averaged out. An arm attached to the nut carries a diamond tool which cuts a secondary helix on the second half of the bar yielding a thread which gives spectra entirely free from ghosts. This arrangement solved the problem of resolving power but not that of photometric efficiency. The latter, defined as accurate control of the distribution of energy in the various diffracted orders, depends on the shapes of the grooves cut. These should preferably be of a right-angle saw-tooth formation in section. Accuracy in this respect depends on the tool used and the material into which the grooves are cut. The tool normally used is an octahedral crystal of diamond which in the course of time may produce a slightly rounded-off curvature at the bottom of the groove but in the normal case this will not affect the groove spacing. But, in cutting the helix groove the material cut loose is pushed into the ridges of the groove and tends to deform the groove nearest to the one in the process of being cut. This has been overcome by exerting a pressure on the cutting tool in the direction away from the grooves already cut. At the time of writing the present article gratings were produced from continuous grooves of up to 3 inch. total length but a new machine was in the process of construction which would permit the use of 4 inch. diameter cylinders cutting helices of up to 30,000 grooves per inch, the aim being to produce a ruled area of 1 sq. ft.

Sommaire : Une description est donnée de la production de grilles optiques par la méthode de Merton.

Article by L. A. Sayce *Endeavour* **48**, Oct. 1953 210-216

Refinements in the Preparation and Theory of Multilayer Films 28/I

United States. It is known that the resolving power of the Fabry-Perot interferometer can be improved to such an extent that it is limited only by the degree of flatness of the plates used. This improvement is effected by replacing the metal films normally used by dielectric films consisting of several superimposed quarter-wave layers of alternatively high and low refractive index. The author prepared nine-layer films of zinc-sulphide and cryolite by vacuum evaporation on crystal quartz Fabry-Perot plates, one pair of which had a flatness of better than $\lambda/50$. The films were tested by comparison with an existing interference filter deposited on glass and consisting of two nine-layer films with a $\lambda/2$ cryolite spacer film in the centre. The pass band of the author's filter was at λ 5,100, its maximum transmissivity was 0.45 and its half-width 15.4 Å. The performance of the filter can be assessed theoretically by calculating the data of the band to be expected on the basis of the electromagnetic theory. Accordingly reflectivities and phase shifts of the 9–layer films as seen from the cryolite spacer were calculated by a matrix method. Then, with the help of the Airy formula (taking phase shift into account) transmissivity was plotted against wavelength. The calculated values were in good agreement with the observed values except for the half width. But when the value of the refractive index of cryolite was raised to 1.4 the calculation gave a half width of 15.8 Å. The value of the absorptivity of the films could be calculated from the peak transmissivity of the filter expressed by $T/(1-R)^2$, where $T=1-R-A$, R and A being the reflectivity and absorptivity respectively of one of the nine-layer films. The calculation showed that, viewed from air, a nine-layer film on glass has a reflectivity of 0.979 at the wavelength at which the reflectivity is maximum, and an absorptivity of 0.007. If the plates of a Fabry–Perot interferometer covered with films of this type were flat to better than about $\lambda/250$, they would have a resolving power of 5×10^6 at λ 5,500 with a spacer of 1 cm.

Sommaire : L'exécution optique de films multi-couches du type employé dans l'interferomètre Fabry-Pérot a été examinée.

Letter by J. M. Stone *J. Opt. Soc. Amer.* **43**, Oct. 1953 927-928

Vol. 4 195

I — General Science and Engineering — I
Contd.

Abstract No. and References

Reflectivity of Thin Aluminium Films and Their Use in Interferometry

29/I

United Kingdom. Fabry-Perot etalons coated with silver or multi-layers of dielectric materials are wholly satisfactory in visible light but not in the region of ultra-violet light. Here, evaporated aluminium films give better results but little is known on the performance of aluminium films with a transmission of 2–20%, typical of the requirements in interferometry. The authors investigated the reflectivity of aluminium films in the region of 4,200 to 2,600 Å. The experimental equipment was similar to that used by Kuhn and Wilson previously (*Proc. Phys. Soc.* **63B** 1950, 745) but the mercury diffusion pump was more powerful. The aluminium used was 99.99% pure. Four flat silica plates, some 25 cm away from the filament, were coated simultaneously in each run. The operational pressures never exceeded 5×10^{-5} mm. Hg. Transmission and reflection was measured by the same method as that described in the previous publication but an ultra-violet sensitive multiplier was used and the light source was a hydrogen discharge tube. The measurements were taken at 4,200 Å, 3,600 Å, 3,200 Å and 2,600 Å, in a band of about 300 Å in each case, transmitted by filter combinations described in detail. Generally, the authors found the previously established fact confirmed that the light transmission of the films increases as the wavelength decreases. Detailed quantitative results are given in graphs showing transmission plus reflection as a function of transmission for each of the four wavelengths. All measurements were taken a few days after evaporation. Further ageing did not seem to alter the performance of the films. The authors observed that slowly deposited aluminium films have lower reflectivity values. This had been noticed by previous workers and was attributed to differences in the crystalline structure of the deposits but the present authors believe to have proved that the phenomenon is due to residual gas occluded in the metal during evaporation. The amount of gas occluded depends on the relative rates of bombardment of the substrate by gas and metal atoms. Thus the reflectivity variations are a function of the product of the processing time and the operational pressure. It appears, however, that there is a lower limit for the values of this product, beyond which this interpretation of the conditions no longer applies. The performance of a Fabry-Perot etalon coated with an aluminium film is given in a further graph showing the effective number of beams, *i.e.* the resolving power divided by the order, on one ordinate, and the intensity relative to the intensity in the absence of an etalon in the maxima of the fringes obtained with monochromatic light, on the second ordinate. This graph demonstrates that thin films with a transmission exceeding 6% have a better performance in the ultra-violet than in the visible region. In conclusion, experiments by a different method are mentioned checking on the performance of aluminium films at wavelengths below 2,600 Å.

Sommaire : Peu de choses sont connues sur les propriétés des couches d'aluminium du type employé dans les interferomètres Fabry-Pérot, lorsqu'elles sont employés dans la région de l'ultra-violet. Des recherches donnant une répartition quantitative de leurs performances sont décrites.

Article by J. C. Burridge H. Kuhn & A. Pery, *Proc. Phys. Soc.* **66B**, Nov. 1953 963-968

The Photo-Electric Effect of Thin Bismuth Films

30/I

See Abstract No. : 20/III

Photoconductivity of Indium Selenide

31/I

United States. Three methods for the preparation of photoconductive indium selenide layers are described. In all cases the substances were evaporated at about 10^{-5} mm. Hg on to graphite (Aquadag) electrodes painted on microscope slides :—(*a*) Indium and selenium were reacted in an argon atmosphere to form a compound of stoichiometric proportions. The compound was then evaporated from a tungsten source but the compound is liable to decomposition on heating and thus no reproducible results can be obtained. (*b*) Indium was deposited first then the support was heated and selenium evaporated on top of the warm indium layer. This method gave inconsistent results. (*c*) The selenium was deposited first and the indium evaporated on top. Then the support was heated to about 200°C, during which period the resistance of the deposit rose to a certain stable value. Adding more selenium (by evaporation) the resistance of the compound layer was increased until further additions produced a reduction of the resistance. If evaporation was stopped at this point the sensitivity of the films showed a maximum. This method gave the most consistent results and films with a sensitive area of 2×10 mm² prepared in this manner had resistance values varying from 0.1 to 100 megohm. The spectral response of the films was measured over the whole range of the spectrum. A monochromator was used in the ultra-violet and a spectrometer in the infra-red. The incident energy was measured with a calibrated thermocouple. The film was connected in series with a battery and a matching resistor and the voltage produced in the films were amplified. The films were tested at 20°C and at –78°C in the latter case the film was in an evacuated chamber during measurement. The spectral distribution of a typical film is shown in a graph, in terms of signal-to-noise ratio per microwatt of incident radiation. It was found that the sensitivity at various wavelengths could be adjusted by changing the proportion of indium and selenium in the film. The time constants of the films were also studied. The photoconductive performance of the films generally indicated that the layers were inhomogeneous. This was confirmed by examination in the microscope. The indium layers consisted of separate crystals and the initial selenium deposit had reacted with the indium forming indium selenide shells around the individual indium crystallites. The second selenium deposit was unreacted. Indium selenide films represent sensitive and versatile detector elements in the ultra-violet, visible and infra-red regions.

Sommaire : Différentes méthodes de préparation et propriétés des couches photoconductive de seleniure d'indium sont décrites.

Article by D. E. Bode & H. Levinstein *J. Opt. Soc. Amer.* 43, Dec. 1953 1209-1210

Thin Films of Ferro-Magnetic Materials

32/I

See Abstract No. : 9/III

I — General Science and Engineering — I

Contd.

Abstract No. and References

A Study of the Propagation Mode for Metallic Vapours in Shadow-Casting by Vacuum Evaporation of Au^{198} and Cr^{51} 33/I

United States. Shadow-casting as used for the preparation of electron microscope specimens is still essentially an art. The thickness of the deposit governs the quality of the shadowing effect obtained in the electron microscope. Normally in shadowing a known mass of metal is evaporated, the dispersion of the material is assumed to be symmetrical and the thickness of the deposit is supposed to follow the inverse square law. In practice, however, considerable deviations of actual density values from those predicted were noticed in many cases. The author investigated these conditions methodically using the following tagged metal tracers as a means of determining the film thickness obtained from evaporation sources of various geometries :—Gold (Au^{198}), typical for a metal wetting tungsten, and chromium (Cr^{51}), typical for a metal not wetting tungsten. Four types of crucibles were investigated : (*a*) A conical wire crucible, (*b*) a cylindrical wire crucible, (*c*) a ' V '–shaped wire source and (*d*) a straight wire source. The vacuum chamber was a bell-jar 30×44 cm. The targets were copper discs, placed in some experiments in the horizontal plane of the crucible, and in other experiments, on the baseplate of the bell jar. Heating of the crucible was strictly controlled and the pressures never exceeded 10^{-4} mm. Hg. The accuracy of the thickness determinations was ±2%. In a preliminary experiment, a source of type (*a*), a seven–turn 30° conical crucible made from 0.025 inch tungsten wire in a perpendicular position with the open base pointing upwards, was surrounded by 10 vertical discs level with the crucible, at various distances. For this arrangement the inverse square law predicts a straight line in a plot of thickness *versus* distance of the substrate. However, evaporating chromium, large deviations occur indicating interference of the crucible structure with the distribution. Evaporating gold in the same arrangement, close agreement with prediction is obtained indicating that wetting of the crucible by the processed material eliminates interference by the crucible structure. But if the crucible position is changed so that its axis is in the horizontal plane the thickness of the gold deposits on targets facing the open end of the crucible is appreciably larger than predicted. Similar conditions were met during deposition at oblique angles, typical of the shadow casting technique, and thicknesses were obtained varying in places by a factor of 10 from the predicted value. A thorough study was then undertaken of the actual distribution from a crucible (*a*) with its axis in the horizontal plane. The nodule of material to be evaporated was held by the three centre turns of the crucible. These three turns were assumed to be wetted, while evaporating gold, and non-wetted while evaporating chromium. It was found, that in both cases the greatest vapour flux issues through the open base of the cone, in the direction 0°. In the case of gold the non-wetted tungsten turns produce a number of umbrae and penumbrae which reduce the emission in all other directions appreciably below that predicted by the inverse square law, particularly so in the direction of 180° (the apex of the cone). In the case of chromium evaporation, the distribution is similar except in the 180° direction where the predicted amount is emitted. But through the open base about 5 times as much as predicted is issued. Crucibles of the type (*b*) give an emission appreciably larger than the inverse square law in both directions of the axis of the cylinder and appreciably smaller at 90° and 270° to the cylinder axis. The only difference between the two metals in this case was that gold gave a wider beam in both directions than chromium. The nearest approach to a spherical distribution pattern was obtained from sources (*c*) and (*d*). Source (*c*) with the ' V ' in the horizontal plane gave the predicted thickness of deposit in the direction opposite to the point of the ' V '. In all other directions the amount emitted was below the inverse square law figure. In the case of source (*d*) a twin-lobed distribution pattern was obtained with an emission approaching the predicted values in the directions perpendicular to the wire axis and with heavily reduced emission in both directions of the axis of the wire. A table reproduced below gives the results obtained from the evaporation of 57.13 mg. of chromium from a 90° conical crucible in the vertical position and targets placed on a plane 6.2 cm below the crucible facilitating the determination of thicknesses at shadowing angles ranging from 25–55°.

Shadowing-Film Thicknesses Obtained with Chromium (Cr^{51})

Evaporation	*Shadowing Angle**	*Predicted thickness by inverse-sq. law*	*Actual film thickness on substrate (by tracer technique)*	*Per cent. of predicted thickness*
1 Cr^{51}	Arc tan 6.2/13 ~25°	136A	37A	27%
2 Cr^{51}	Arc tan 6.2/10 ~30°	250A	76A	30%
3 Cr^{51}	Arc tan 6.2/7 ~40°	632A	154A	24%
4 Cr^{51}	Arc tan 6.2/4 ~55°	1040A	271A	26%

* Arc tan Crucible height/Horizontal target distance.

Through the whole range the thickness of the deposits was about a third of that predicted by empirical formulae. The effects obtained from these deposits in the electron microscope are demonstrated in reproductions of electron micrographs indicating that the optimum value lies between 37 and 76 Å thickness.

Sommaire : Une étude méthodique de distribution obtenue par des sources d'évaporation de géomètries différentes, un problème d'une signification particulière sur les effects obtenues par la mèthode de projection d'ombres.

Article by L. E. Preuss *J. Appl. Phys.* **24**, Nov. 1953 1401-1409

Absorption Coefficients of Nitrogen Oxide in the Vacuum Ultra-Violet 34/I

United States. It is believed (but not yet finally established) that the upper atmosphere contains nitrogen oxide (NO) and some workers suggest that its photo-ionisation by the sun's radiation in the range 1,100–1,300 Å

Vol 4 195

I — General Science and Engineering — I

Contd.

Abstract No. and References

accounts for the formation of the D–layer. Nitrogen oxide can be produced photo-chemically but the mechanism of its formation is not fully understood. It is important to obtain quantitative absorption data for this gas in the range 1,100–1,300 Å in order to assist exploration of the above mentioned conditions. The present article reports on an investigation of the absorption spectrum of nitrogen oxide in the range 1,100–2,300 Å. The experimental arrangement generally and in particular the light source, vacuum monochromator and detector are the same as described by Watanabe and Inn (*J. Opt. Soc. Amer.*, **43**, 1953, 32). The absorption cell was 4.7 cm long, fitted with lithium fluoride windows and positioned between the photo-multiplier detector and the exit slit of the monochromator. Two McLeod gauges, a mercury manometer and an ion gauge were used for pressure measurement. The monochromator chamber was held at about 2×10^{-4} mm. Hg, and the nitrogen oxide supplied to the absorption cell was kept at any pressure level between 0.3 and 100 mm. Hg as required for the various experimental runs. For the measurements in the Schumann range the nitrogen oxide supplied was subjected to repeated fractional distillation in order to obtain the required purity, *i.e.* 99.95%. In the region 1,350–2,300 Å the spectrum was found to consist of several systems of sharp bands including the β, δ, ϵ and γ bands. Above 1,900 Å some overlapping occurs, but at shorter wavelengths the spectra are more complex. The absorption intensities obtained for the range 1,500–2,300 Å during a run at 14.8 mm. Hg are shown in a graph. These results are compared with those of other workers. Certain possible continua are indicated in a plot of the values of the absorption coefficients (k, in cm^{-1}) covering the whole range considered and the absorption coefficients for several minima in the region 1,330–1,515 Å, determined at different pressures, are tabulated. At the Lyman alpha line of hydrogen (1,126 Å) the value of k is 67.5 cm^{-1}. The continua traced in this investigation permit the tentative conclusion that photo-ionisation takes place on large enough a scale to facilitate the formation of the D-layer.

Sommaire : Les données d'absorption de NO furent determinées dans la région de 1,000 à 2,500 dans le but d'obtenir des informations essentielles sur la partie qui ce gaz pourrait jouer aux plus hautes régions de l'atmosphère.

Article by F. F. Marmo *J. Opt. Soc. Amer.* **43**, Dec. 1953 1186-1190

Cosmic Ray Production of Helium in Meteorites and their Ages — 35/I

See Abstract No. : 53/III

The Liquid Helium Plant of the Physical Laboratory of the University of Louvain — 36/I

See Abstract No. : 35/II

Metal-to-Glass Seals for Sub λ Point Helium — 37/I

See Abstract No. : 17/IV

Boiling Point of Capillary-Condensed Water — 38/I

India. A simple method has been devised for the determination of the boiling points of capillary-condensates. The capillary system containing the liquid to be examined is placed in a small chamber which is evacuated until a differential manometer registers the vapour pressure of the liquid. An oil bath surrounding the chamber is then heated until the vapour pressure is equal to atmospheric pressure. This temperature of the bath is identical with the boiling point to be determined. By this technique the average values of the boiling point elevations of water condensate in pores of silica-gel, sugar, charcoal, bentonite and clay loam have been found to be about the same, as long as the capillaries have the same radii. The values obtained for water ranged between 2.4°C and 34.1°C for pores of radii 1,060 Å to 9 Å respectively and agreed with the values calculated from thermodynamic considerations based on the theory of capillary condensation.

Sommaire : Une méthode simple pour mésurer le point d'ébullition de l'eau condensée dans les pores de matériaux poreux est décrite.

Letter by M. L. Lakhanpal & B. R. Puri *Nature* **172**, 14.11.53 917

Rate of Evaporation of Glycerol in High Vacuum — 39/I

United States. The rate of evaporation from the surface of a liquid in contact with its equilibrium vapour is derived from the kinetic theory and is given by $r = p\sqrt{M/2\pi RT}$, where r is the mass per unit area per unit time, p is the vapour pressure at T° absolute temperature, M is the molecular weight and R is the gas constant. As practical evaporation rates did not reach the values calculated, a factor, the ' evaporation coefficient ', was introduced to take care of the difference in each case. In a series of publications (see Abstract No. 208/III, Vol. No. II) experiments are described which demonstrated that, if the surface of the liquid is truly representative of the bulk and kept free from contaminations, evaporation takes place at the maximum theoretical rate, *i.e.* the evaporation coefficient is unity in all cases, irrespective of the nature of the processed material. This thesis was supported by experimental proof but as these experiments dealt only with two types of materials, the phthalate and sebacate esters, it was decided to make a further series of experiments with a material of exceptionally low vapour coefficient. The present article reports the procedure and results of these experiments. Glycerol was the material chosen. It has a reported evaporation coefficient of 0.52 at 18°C with a vapour pressure less than 0.1 micron. The apparatus used was a falling stream tensimeter substantially the same as that

I — General Science and Engineering — I
Contd.

Abstract No. and References

described in Abstract 206/III, Vol. II but specially adapted for the purpose. The glycerol was prepared by distilling off its water content. The 8 middle fractions, obtained in distillation, only were used. 550 ml. of the de-watered glycerol were degassed in the tensimeter which was evacuated by a vapour diffusion pump backed by a mechanical pump. The glycerol was circulated through the apparatus by 2 centrifugal impellers in series. A cylindrical wire screen positioned just before the jet tube entrance distributed the glycerol radially. Acetone at –70°C was circulated through a jacket to cool the zone for the collection of condensate. At the end of a run the frozen condensate was melted by switching off the diffusion pump and the acetone supply, replacing the latter by a stream of air of room temperature. The condensate was then withdrawn by the mechanical vacuum pump and weighed or returned to the main bulk of the fluid. Drainage time varied between 1 hour for 70°C runs to 1½ hours for runs at 30°C. In order to measure the evaporation rate at 18°C the apparatus was modified. Larger tubes were fitted to avoid pressure drop and the liquid was circulated by a screw lift in the form of a copper tubular spiral rotated by an external motor through a shaft seal. The glycerol was cooled by water at 10°C, passed through a jacket surrounding the screw lift. The condenser temperature was –67°C. One run took 4 days and draining 3 hours. Measurements in the condensing zone were carried out photographically. The range of measurement and the results are shown in a table partly reproduced below, indicating that, within the limits of experimental error, the evaporation coefficient was unity over the whole range.

Comparison of Observed and Calculated Values for Rate of Evaporation of Glycerol

Temp. °C	*Vapor Pressure, Microns*	*Rate of Evaporation, g./sec. sq. m.*		*Difference between Obsd. and Calcd. Values of Rate, %*
		Obsd.	*Calcd.*	
70.1	17.0	4.83	5.14	— 6.0
58.5	5.8	1.79	1.78	+ 0.6
50.4	2.58	0.892	0.805	+10.8
38.2	0.72	0.244	0.228	+ 7.0
18.2	0.069	0.0261	0.0226	+15.5

Sommaire : Glycérol est signalé comme ayant un coéfficient d'évaporation très bas. Afin de prouver que la théorie Torpide s'applique à tous les matériaux, le glycérol a été soumis à des tests qui montrèrent une valeur du ' coefficient d'évaporation ' égal à l'unité, après élimination des impurités et l'obtention d'une surface propre.

Article by D. J. Trevoy *Industr. Engng. Chem.* 45, Oct. 1953 2366-2369

16 — CHEMISTRY — 16

The Preservation of Arterial Grafts by Freeze Drying

40/I

United Kingdom. The surgical technique of blood vessel grafting has been known for nearly 50 years. In the last 6 years this practice has been extended to the treatment of human patients particularly in the following cases : Aneurysms, wounds, primary thrombosis, after some emboli, in certain cases of malignant diseases and in isolated cases of arteriosclerosis. The increased use of this technique required the establishment of so-called artery banks. In the early days the arteries were preserved in 4% formaldehyde at a temperature just above freezing point. It is now known that this technique is inferior to frozen or freeze-dried vessels so far as the period of safe storage is concerned. Freezing the arteries at temperatures of –20°C to –80°C improves the safe storage period but causes problems in transport. These remaining difficulties have been overcome by applying the freeze drying technique to the preservation of arteries. Details of the method employed by the author are given. The arteries are placed in a sterile Pyrex tube and frozen to –79°C. The tube is then evacuated to 0.05 mm. Hg and kept under vacuum for approximately 9 hours. During this period the moisture sublimes but the artery remains cool due to the loss of the latent heat of evaporation. No external cooling is needed owing to the small volume of the processed material. The moisture sublimed is removed by condensation and/or trapped in phosphorus pentoxide. At the end of the 9 hours the graft has lost about 95% of its original moisture content and begins to warm up to room temperature. The graft is then transferred to a desiccator fitted with a charge of phosphorous pentoxide. The desiccator is evacuated and the graft remains in the apparatus for about 4 days at room temperature. In this manner the moisture content of the graft is reduced to 1–2%. The tube containing the graft is removed from the desiccator, closed and evacuated with the help of a hypodermic needle. In this condition the graft can be stored for many years at room temperature and can be carried by the surgeon in his instrument bag wherever he goes. Immediately prior to the use of the artery the test tube, still under vacuum, is filled with sterile isotonic saline and after about 30 minutes the artery is completely re-hydrated. The technique has the practical disadvantage that the donor must be under 25 years of age and the theoretical disadvantage that the tissue is dead. In the personal experience of the author freeze-dried arterial grafts were successful in 7 out of 9 cases.

Sommaire : Description brève d'une méthode de lyophilisation des artères pour un long stockage et indications sur les emplois médicaux des artères ainsi traitées.

Article by C. G. Rob *Proc. Roy. Soc. Med.* 47, No. 5, 1953 16-18

I — General Science and Engineering — I
Contd.

Abstract No. and References

Electrical Potential Differences Across the Normal Aorta and Aortic Grafts of Dogs

41/I

United States. The differences observed in the performance of fresh and freeze-dried grafts led to an investigation, reported in this article, of the values of the electrical potential differences across the aortic wall of both types of grafts in order to obtain more information on the causes of these differences. Calomel half-cells were used as electrodes joined to polyethylene catheter tubing fitted with decinormal sodium chloride solution serving as the ionic conductor. One electrode was connected to the adventitia (outer coat of the aortic wall) and the other to the intima (the innermost coat of the aortic wall). The experiments were carried out on dogs. A.o. the following conditions were studied : The normal aorta, grafts of fresh aorta before and after transplantation and grafts of freeze-dried aorta before and after transplantation. Some long term experiments extended over several weeks. The normal aortic wall was found to have a p.d. of the intima with respect to the adventitia varying from –0.5 to –5.0 mV. If the aorta is injured, the polarity may change temporarily. All fresh grafts had a potential and the p.d. was close to that of the normal aorta. After implantation, polarity changes and oscillations of p.d. values occurred temporarily followed by normal polarity and consistent p.d. values during the final healing period. Freeze-dried grafts show no p.d. after reconstitution. But a fortnight after implantation a very low p.d. of normal polarity could be measured. The electrical data obtained have been proved to be strongly indicative of the histological changes such as progressive ingrowth of connective tissue and physiological effects resulting from aorta implantations. The electrical performance of fresh grafts is correlated to thrombosis. No thrombosis is indicated in the case of freeze-dried grafts. The presence of intima could be consistently demonstrated in fresh grafts soon after implantation but never in freeze-dried grafts.

Article by P. N. Sawyer & J. W. Pate *Amer. J. Phys.* 175, Oct. 1953 113-117

Sommaire : Le mesure de différences de potentiel existant entre les parois d'aortes normales et d'une aorte greffée, fraîche ou lyophilisée, donne une indication des changements histologiques et physiologiques prenant place après l'opération.

A Study of the Factors Governing the Rate of Freeze Drying of Blood Plasma

42/I

United Kingdom. The rate of drying frozen blood plasma has been studied in a laboratory apparatus with a view of obtaining data useful for the assessment of the performance of commercial plant. The apparatus used is similar to one described previously (see Vol. III Abstract No. 142/I) and employed in studies of the evaporation of pure ice and the drying of frozen clay. The latter is typical of the conditions applying to the drying of frozen blood plasma but presents less difficulties in investigations of the laws governing the process. The rate of evaporation from a surface of pure ice, situated in an evacuated chamber, a few centimetres away from a large condenser kept at comparatively low temperature is expressed by

$$G = 60^2\gamma \sqrt{\frac{M}{2\pi RT}} \cdot P_0 = \gamma K P_0 \qquad (1)$$

where γ is a constant near unity, K a function of temperature only and P_0 the vapour pressure of the ice at the operational temperature. The order of magnitude of the evaporation rate is demonstrated in a table, partly reproduced below :

Table I

Temp. °C.	*P_o mm.*	*G cm. per hour*
0	4.6	150
–30	0.29	10
–50	0.029	1.73
–60	0.008	0.336

In the above table the rate is expressed in terms of retreat of ice surface (in cm) p. hour. If frozen clay with 50% moisture content is dried in the same way, the retreating ice surface leaves a gradually growing dry layer of clay on top of the moist clay which offers resistance to the escaping water vapour. In this case the rate of evaporation is expressed by :

$$G = \frac{1}{\rho} \frac{P_u^2 - P_d^2}{L} \qquad (2)$$

where P_u is the upstream pressure, P_d the downstream pressure, L the thickness of the dried material and ρ the specific flow resistance. The latter is an equivalent of the specific resistance in the electrical theory. Its dimension is

$$\frac{cm^2 \times hr. \times (mm.\ Hg)^2}{g}$$

In the experiments with blood plasma a solution of 1 g. of dried blood plasma in 12 g. of distilled water was used. Details of its composition are given. As the resistance of the dried plasma to the escaping vapours was very much less than that of clay the heat input could no longer be adjusted to give a constant ice surface temperature. Instead, the heat input was kept constant resulting in the ice surface temperature increasing as

I — General Science and Engineering — I
Contd.

Abstract No. and References

the drying continued but the rate of evaporation was remarkably constant throughout the run and could be expressed by

$$G = \frac{1}{\rho}\frac{P_o^2}{L} \qquad (3)$$

This is in fact the same law as that expressed by Equ. 2 because $P_o = P_u$ and $P_d = 0$ owing to the comparatively low temperature of the condenser. The values for ρ derived from this expression are fairly constant as is demonstrated in the following table giving the results of a number of runs at various heat inputs :

Table II

G	*Temperature* Initial	Final	$\rho = \frac{P_o^2}{GL}$	*average* ρ
0.26	−29	−23	1.93 1.19 1.23 1.52	1.47
0.216	−34	−23	1.50 1.31 1.59	1.47
0.336	−30	−20	1.56 1.93 1.74 2.40	1.91

The particular large-scale process, to the assessment of which the above findings have been applied, is the method described by Greaves in H.M.S.O. Special Report Series No. 258 (1946). The essential details of the method are described and shown in illustrations. The respective formulae are developed and the maximum permissible temperature of the layer of the processed material nearest to the heater is calculated for the drying rate quoted by Greaves in order to compare it with the quoted value of the observed ice temperature. The calculated value is −23°C. This figure agrees with practical measurement. The rate of evaporation from an unimpeded ice surface at −23°C to a condenser at −40°C is of the order of 15 linear cm. of ice per hour but the evaporation rate observed in the plant is only about 0.05 cm./hr. Further comparison shows that the resistance to evaporation offered by the dried plasma is about five times that of the filter used on the bottles. Higher rates of evaporation could be achieved by developing designs eliminating the need of a filter and providing for the finished dried plasma to fall away from the ice surface. Difficulties in applying adequate heat to maintain higher evaporation rates without raising the temperature above the permissible limit may be overcome by treating very thin layers of material only or by applying radiant heat.

Sommaire : Les facteurs essentiels gouvernant les performances d'un appareil à lyophiliser le plasma sanguin sont investigués en étudiant premièrement la sublimation de glace pure et deuxièmement la lyophilisation d'argile contenant 50% d'humidité.

Paper by H. A. Kearsey R. F. Strickland & E. W. Bruce *Preprint Inst. Refrig.* Nov., 1953

The Behaviour of Light-Sensitive Evaporated Layers in the Electron Microscope 43/I

See Abstract No. : 19/II

The Activities of Evaporated Metal Films in Gas Chemisorption 44/I

United Kingdom. Experience with metal catalysts has shown that the best results for many reactions are obtained with transition metals. This has given rise to the theory that the chemical bond between metal and adsorbate involves the metal d-band. This theory has been confirmed by experiment in several instances where transition metals of the group VIII of the Periodic Table have been used. But nothing is known about the respective properties of the other metals. For this reason, in the present investigation about twenty different metals, in the form of evaporated films, have been studied with regard to their chemisorption activities when exposed to hydrogen, nitrogen, oxygen, carbon monoxide, ethylene and acetylene gases at various temperatures. The apparatus used and the methods by which the gases (except acetylene) were prepared have been described previously (*Proc. Roy. Soc.*, A, **205**, 1941, 409; *Trans. Fara. Soc.*, **48**, 1952, 160.) Most of the metals were of a purity better than 99.95%. Nickel, rhodium, molybdenum and silver were evaporated from filaments of the metal itself. Potassium films were obtained by a distillation procedure. All other films were deposited by evaporation from tungsten wire sources. The tungsten filaments were outgassed to 2,800°C before use and the metals to be deposited were outgassed up to their evaporation temperature. The table reproduced on the following page summarises the results of this investigation.

Vol. 4 1954

I — General Science and Engineering — I
Contd.

Abstract No. and References

Group	*Metals*	*Gases*					
		Nitrogen	*Hydrogen*	*Carbon Monoxide*	*Ethylene*	*Acetylene*	*Oxygen*
A	Tungsten, Tantalum, Molybdenum, Titanium, Zirconium, Iron, Calcium, Barium.	+	+	+	+	+	+
B	Nickel, Palladium, Rhodium, Platinum.	—	+	+	+	+	+
C	Copper, Aluminium.	—	—	+	+	+	+
D	Potassium.	—	—	—	—	+	+
E	Zinc, Cadmium, Indium, Tin, Lead, Silver.	—	—	—	—	—	+
F	Gold.	—	—	+	+	+	—

The metals are divided into six groups, A–F, in order of decreasing activity. The experiments prove that the affinities of the gases to the metal surfaces investigated can be graded as follows :

Oxygen > Acetylene > Ethylene > Carbon Monoxide > Hydrogen > Nitrogen

A metal which chemisorbs one gas, will chemisorb all gases higher in the scale and *vice versa* if it does not chemisorb a particular gas, it will not chemisorb gases lower in the scale. The one exception is gold. The highest activities are shown, with a few exceptions, by the transition metals or metals having the characteristic partly filled d–band of the transition metals, represented by groups A and B. Thus chemisorption of all gases studied appears to require d–band vacancies. But this does not apply to oxygen. Oxygen chemisorption occurs on both transition and non-transition metals. In this case, chemisorption seems to take place with the formation of 0^{2-} ions, whereby the electrons are supplied from the s– or p–band of the metal, rather than the d–band. However, the behaviour of gold cannot be explained satisfactorily by this theory.

Sommaire : Des expériences ont permis d'étudier le mécanisme de chimisorption sur environ soixante-dix métaux différents, exposés, sous forme de films evaporés, à différents gaz.

Article by B. M. W. Trapnell *Proc. Roy. Soc.* A, **218** 23.7.1953 566-577

Sorption Properties of Thin Nickel Films

45/I

Germany. An experimental and theoretical study of the sorption of oxygen and hydrogen by evaporated films of nickel. Details of the apparatus and methods are given. The sorption of oxygen can be considered as two processes, (*a*) the surface of the metal film is first covered momentarily by a monatomic layer of oxygen; (*b*) the oxygen atoms are then forced into the interior of the film. (*a*) Is independent of the gas pressure in the sorption apparatus. Hydrogen is not so strongly absorbed by nickel but nickel which has previously been exposed to oxygen behaves differently towards hydrogen, the sorption now being very much greater. Previous exposure of nickel films to hydrogen does not influence the subsequent sorption of oxygen.

(*Science Abstracts*)

Sommaire : Les propriétés de sorption de films fins de nickel ont été étudiées vis à vis de l'oxygène et de l'hydrogène.

Article by W. Scheuble *Z. Phys.* 135, No. 2, 1953 125-140

The Solubility of Gases in Lubricating Oils and Fuels

46/I

United Kingdom. Oil or fuel employed in aircraft show excessive gas and vapour evolution when the plane reaches a certain height. The actual altitude at which this phenomenon occurs may vary with the relevant properties of the material, *i.e.* with its vapour pressure and the solubility coefficient of air in the material. So far as (low viscosity) aviation fuels are concerned an accurate method for the determination of the solubility of gases in the material is already in existence (see R.A.E. Farnborough, Report No. Chem. 464, 1950) but little is known on the properties and performance of (high viscosity) petroleum oils in this respect. In the present paper a method is described which facilitates gas solubility measurements on petroleum oils. This group of materials has a negligible vapour pressure. In the new method the oil sample is fed into an evacuated vessel in order to liberate and remove all dissolved air. This process consists of three steps : Degassing—intermediary

I — General Science and Engineering — I
Contd.

Abstract No. and References

saturation—degassing. Then the oil is passed to an evacuated displacement burette where complete saturation with the gas under examination is effected by bubbling the gas through the oil. Finally, the oil is transferred by means of a Toepler Pump to a vessel of calibrated volume held at 10^{-3} mm. Hg, where the amount of gas taken up by the oil in the saturation process is released and determined. Details of the procedure are given. The results of experiments, carried out at 20°C are shown in a table, partly reproduced below.

Solubility Coefficients of Atmospheric Gases in Oils and Fuels

Temperature, 20° C; partial pressure, 760 mm. Hg		*Carbon dioxide*		*Oxygen*		*Nitrogen*		*Air (dry and carbon dioxide free)* 80% *Nitrogen* 20% *Oxygen*		*Air calcd. (dry and carbon dioxide free)*
Liquid	*Viscosity at 100° F., cs*	α_v	γ	α_v	γ	α_v	γ	α_v	γ	α_v
Oil A.1 – –	615	0.853	0.915	0.124	0.133	0.0662	0.0711	0.0783	0.0840	0.0782
Oil A.2 – –	268	0.861	0.924	0.126	0.135	0.0664	0.0713	0.0792	0.0850	0.0789
Oil A.5 – –	34.9	0.966	1.037	0.145	0.155	0.0762	0.0818	0.0903	0.0969	0.0905
100-octane fuel C	– –	—	—	—	—	—	—	0.243	0.261	—
Water – –	– –	—	—	—	—	—	—	0.01858 0.01875	—	—

The properties and compositions of the materials tested are given as follows:

Properties of Oils

Oil	*Sp. Gr.* 15°/4° C	*Flash point (closed)* °F	*Kinematic viscosity, cs.* 100° F	210° F	*V.I.*	*Pour point* °F
A.1 (Residual)	0.894	510	615	34.4	95	0
A.2* (Residual)	0.885	460	268	20.3	96	0
A.5 (Distillate)	0.869	350	34.9	5.4	96	0

* Conforms to DTD 472B Specification.

Composition and Properties of Fuels

Fuel	*Composition*	*Hydrocarbon analysis*	*T.E.L., ml/ I.G.*
100–octane fuel C	40% Hydrocodimer 43% Hydro-base 3% Straight run 14% *iso* Pentane	2% Olefins 2–3% Aromatics 22% Naphthenes 73–74% Paraffins	4.0

α_v is the Bunsen coefficient, defined as the volume of gas at N.T.P. dissolved in unit volume of the liquid, when the gas partial pressure is 760 mm. Hg and is expressed in terms of Henry's law : $\alpha_v = 760$ K. γ is the Ostwald coefficient which can be regarded to express as above the volume of gas dissolved in unit volume of liquid but the volume of gas is measured, not at N.T.P., but at the experimental conditions of pressure and temperatures during the saturation process. It is correlated to α_v by : $\gamma = \alpha_v$ (T/273.2). The data on fuels shown in the table was obtained by the same method but certain modifications to the procedure were required. Results of further experiments are shown giving information on the effects of temperature in the range of 0–100°C. In the case of air, for instance, these experiments have shown that the Bunsen coefficient of the A.2 and A.5 oils remains constant throughout the range of temperatures considered. This is due to the fact that the Bunsen coefficient of nitrogen increases with temperature whereas that of oxygen decreases. As a result of these conditions the Ostwald coefficient rises with temperature. The outcome of the experiments generally is compared with the interpretation of the conditions by the theory proposed by Hildebrand in 1936. Although this theory was originally restricted to gas solubility in pure liquids the authors found that oils and fuels representing mixtures behave similarly to pure liquids and that this theory may be used for the prediction of the respective performance of petroleum oils with certain qualifications.

Sommaire : On décrit une méthode et un équipement permettant la détermination de la solubilité de gaz dans des huiles de pétrole de viscosité élevé, facteur important qui conditionne les performances de ces huiles dans les moteurs d'avion volant à haute altitude.

Paper by R. R. Baldwin & S. G. Daniel *J. Inst. Petrol.* 39, 1953 105-124

Mass Spectrometric Determination of Gas Impurities Separable by Condensation 47/I

See Abstract No. : **2/IV**

I — General Science and Engineering — I
Contd.

Abstract No. and References

Rapid Interferometric Analysis of Germane-Hydrogen Mixtures 48/I
See Abstract No. : 3/IV

Design and Operating Technique of a Vacuum Drying Oven 49/I
See Abstract No. : 23/III

17 — METALLURGY — 17

Production of Titanium Metal 50/I
See Abstract No. : 35/III

Improvements i.o.r.t. the Manufacture of Calcium 51/I
See Abstract No. : 36/III

Very Pure Metals 52/I

United Kingdom. Normally, ordinary metals contain up to 1% impurities. The first initiative to produce really pure metals was most likely prompted by research requirements. For instance, the first highly purified silver was prepared by Stas in 1865 to facilitate the determination of its atomic weight. Since then, pure metals have acquired industrial importance, *e.g.*, as construction materials in atomic energy installations. The fact that a great variety of highly purified metals is available to-day is largely due to the great progress made in vacuum engineering over the last 15 years. Aluminium of commercial grade may contain up to 0.5% of iron and silicon which reduces its corrosion resistance appreciably. Hoopes and Frary, U.S.A., devised an electrolytic process which facilitated the production of aluminium of 99.99% purity. This material is highly corrosion resistant and used for cladding high-strength aluminium alloys employed in aircraft and marine equipment. An alternative method of producing very pure aluminium, employing vacuum, is under development in England. Aluminium chloride is contacted with molten aluminium at 1,000°C under very low pressure. As a result volatile sub-chloride is formed which, on cooling to 600°C, splits into aluminium and the usual chloride. One of the most important vacuum distillation processes used for the purification of metals, has been developed by van Arkel and de Boer. It is employed for the production of pure titanium and zirconium, and in a modified form for the production of pure vanadium, chromium and hafnium. It is based on the fact that these metals form volatile iodides at about 600°C, the iodides being decomposed into the pure metal and free iodine when in contact with a tungsten filament held at 1,200°C (see Vol. III Abstract No. 170/III). In the case of vanadium, the modified van Arkel process is applied to the end product of the refining method, where vanadium pentoxide is reduced with calcium. This end product, in the form of beads, contains about 0.02% oxygen and a certain amount of nitrogen which makes it brittle. The beads are placed in an evacuated chamber and exposed to iodine vapours at 900°C. The iodide is sublimed and contacted with a molybdenum grid where, by means of a filament held at 1,400°C., the iodide is decomposed into the pure metal and iodine. Similar processes involving the use of a suitable compound, for the removal of impurities present in the metal are described for tantalum, silicon, germanium, beryllium, niobium, etc. Other major difficulties are presented by the tendency of certain metals to combine with elements such as oxygen and nitrogen contained in the air. This affects the preparation of pure metals in the form of ingots. In particular, titanium, zirconium, hafnium and vanadium in the molten state will pick up impurities from the atmosphere or from the crucible. Evacuation alone is only a partial remedy. But satisfactory results have been obtained by melting these metals with an arc in a low-pressure argon atmosphere, using a pressed bar of the metal processed as one of the two electrodes. Powder metallurgy techniques are employed to make sheet, rod and wire from hydrogen reduced metal powders of platinum, nickel, cobalt and iron. These methods require heat-treatment of the parts in vacuum at the final stage to remove the last traces of hydrogen and oxygen occluded in the material. In the case of chromium no satisfactory purification method is available yet. Although a high degree of purity can be obtained in most of the metals by modern methods it is still not good enough for certain metals in particular applications. For instance germanium and silicon used in rectifying devices must be of exceptional purity. Details of a method reducing the impurity content of germanium to less than one part in a thousand million are given.

Sommaire : Revue des différentes méthodes pour la purification des métaux. Des détails d'un certain nombre de procédés employant le vide sont donnés.

Article by A. R. Powell *Times Sci. Rev.* Autumn 1953 14-16

Development of Commercial Vacuum Furnaces for Melting Metals and Alloys 53/I
See Abstract No. : 14/I

I — General Science and Engineering — I
Contd.

Abstract No. and References

Metal Melting Under Vacuum. Some Mechanical Problems — 54/I

United Kingdom. During the last ten years the process of vacuum melting has outgrown operations on a laboratory scale. There is now standard equipment available for production on an industrial scale with crucible capacities ranging from 20–240 lb. per charge. One installation in Switzerland, is known to consist of a furnace with a capacity of 450 lb. per charge. Details and illustrations are given of a modern 60 lb. plant. Its vacuum chamber is made of stainless steel and is double-walled to facilitate water cooling. It is fitted with a feeder device of the type described in Vol. III, Abstract No. 125/III. The quartz glass inspection window in the cover is reinforced with a panel of Pyrex glass. Metal films deposited on the window during processing can be removed by a wiper in the form of a wire brush, operated from outside the chamber. Pyrometric measurement is effected by means of a thermocouple housed in a quartz tube. A special mechanism, controlled externally, permits the insertion of the thermocouple into the crucible when required and its withdrawal, when not in use, to an area where it cannot suffer mechanical or thermal damage, during pouring for instance. Various methods of pouring are mentioned. In the plant described the chamber is stationary and the crucible only is tilted for the purpose. Heating is effected by induction and the moulds of cast-iron or steel, have a smooth surface and are pre-heated to about 300°C immediately prior to use. The progress of a typical tool steel melt is shown in a time-pressure diagram, details of which were discussed in Vol. III, Abstract No. 164/III. The pressure in the chamber, before melting starts and immediately prior to pouring, is 10^{-3} mm. Hg. It is stated that the consumption of electrical energy for vacuum melting differs little from that required for melting an equivalent charge in the same type of furnace at atmospheric pressure.

Article by Anon. *Metallurgia* Nov. 1953 259-260

Sommaire : Détails d'un four à induction à vide d'une capacité de 30 kg convenant pour la fusion et la coulée d'aciers pour outil.

Furnace for Vacuum Distillation of Zinc and Silver Contained in Alloys of Zinc and Silver, Constructed by the Institute Pesquisas Tecnologicas of Sao Paulo — 55/I

See Abstract No. : 32/III

The Evaporation of Metals and Metalloids in Vacuum — 56/I

See Abstract No. : 38/III

Method of Preparing Nickel Powder with a Large Surface Area — 57/I

United States. Fine nickel powders are used as chemical, physical or catalytical agents. For instance, it is sometimes necessary to measure the heat produced when nickel powder is wetted with certain liquids. Also, nickel powder is employed in the hydrogenation of vegetable oils for the production of shortenings. For this and other applications it is desirable to make nickel powders available which have a large specific surface. At present, nickel powders obtainable through normal commercial channels have surface areas not exceeding 1 m^2/g. In isolated instances nickel powders with surface areas of 50 m^2/g. have been produced but the processes involved are rather complex. A method for the regular production of large surface nickel powders is described as follows: Nickel oxalate ($NiC_2O_4 . 2H_2O$) is heated in a tube at 200°C in an oven to eliminate the water of crystallisation and then heat-treated at about 320°C. The tube is exhausted in order to accelerate the decomposition of the material and to prevent conversion to the oxide or carbonate. Every 24 hours the tube is closed by means of a stopcock, removed from the oven, cooled down and the contents weighed to check on the progress of the decomposition. When decomposition is 99–100% complete, the powder is ready for use. If the powder is to be stored, benzene or an inert gas is introduced into the tube without breaking the vacuum to protect the material from oxidation. When required for use, the nickel benzene mixture is placed in glass bulbs connected to a vacuum system to evaporate and remove the benzene. Evacuation is continued at 320°C and the bulbs are sealed off when the pressure has reached 10^{-5} mm. Hg. The temperature applied during treatment is important. Nickel powder prepared at 400°C has a surface area of about 44 m^2/g. The powder produced by this method is pyrophoric. In contact with liquids such as benzene it evolves heat of the order of 0.1 to 0.2 cal./m^2.

N. J. DeLollis *U.S. Pat.* 2,660,523

Sommaire : Méthode pour la préparation de poudre de nickel à grande surface.

Vacuum Dezincing of Desilverised Lead Bullion — 58/I

See Abstract No. : 33/III

Production of Pure and Deliberately Contaminated Iron and Studies of These Materials — 59/I

See Abstract No. : 34/III

Vol 4 195

I — General Science and Engineering — I

Contd.

Abstract No. and References

Method of Testing a Metallic Sample

60/I

United States. The known methods of testing metals for certain impurities (other than free gases) occluded in the material are difficult to operate and may not be foolproof regarding the detection of deeply embedded matter. A new method has been developed which is less time consuming and is described in detail with special reference to nickel as the metal under test. It is applicable to any impurity such as nickel, oxide and carbon, which combines with atomic hydrogen to form a volatile compound. The specimen material in the form of a solid cylinder is enclosed by a glass bulb and supported by an earthed electrical lead, sealed through the wall of the envelope. This lead connects to a radio-frequency source, the sample acting as one electrode. A metal cylinder surrounding the glass bulb serves as the second electrode. A tube joined to the bulb leads to a T–junction, the arms of which are connected *via* stopcocks to a vacuum pump and to a source of molecular hydrogen respectively. As a first step occluded gases are liberated by heating the glass bulb containing the metal cylinder in an oven to approximately 400°C and subsequently by heating the sample by induction to 900°C for 1–2 minutes or longer. Then the glass bulb is evacuated. After evacuation the pump is shut off and hydrogen is admitted to the bulb at a pressure of 0.3 to 0.4 mm. Hg. Operating the radio-frequency source (4 kV, 6 Mc/s), the molecular hydrogen in the glass bulb is ionised and the atomic hydrogen thus formed is gradually absorbed by the metal. For this reason more molecular hydrogen must be added at intervals during this operation. After 5–10 minutes the bulb is cooled to room temperature, refilled with fresh hydrogen, sealed off and the specimen is heated again to 900°C by induction heating for 2 minutes. At this stage the volatile compounds which have formed from the occluded impurities and the atomic hydrogen during the ionisation process are liberated. Operating the radio frequency source a glow discharge is produced emitting a colour characteristic of the nature of the impurity present which can thus be visually or spectrographically identified.

Sommaire : Description d'une méthode de detection des impurités (autres que des gaz libres) occlusés dans des métaux, en se référant particulièrement au nickel employé pour les essais.

M. L. Yeater & U.S. Navy *U.S. Pat.* 2,656,256

Vol. 4 1954

19 — SCIENCES (other than the above) — 19

The Influence of Barometric Pressure on Watch and Chronometer Rates

61/I

See Abstract No. : 15/I

Photographic Copying Process

62/I

Germany. The conventional method of producing copies of pictures, scales, etc. on a transparent plate requires the application of a lead sulphide film to the transparent plate which is either engraved or etched accordingly. This method has certain disadvantages. Engraving is a tedious and lengthy operation while the edges of copies formed by etching lack satisfactory sharpness. These disadvantages are overcome in a new process described in this patent. A light sensitive layer, such as bichromated shellac of a suitable composition, is applied to the support which may be glass, metal or plastics and is exposed in part by contact or projection to the original to be copied. After completion of the exposure, the exposed part of the layer is washed out, leaving the surface of the support bare in that area. Then, by means of vacuum evaporation, a thin layer of metal such as chromium is deposited over the remaining parts of the light-sensitive layer and the bare area of the support. Now, the light-sensitive layer with its top layer of chromium is dissolved by known chemical means and removed. As a result, all that remains on the support, is the part of the mirror-like chromium deposit which forms a true copy of the original. The process can be refined by adding at this stage another layer of a suitable material by vacuum evaporation and dissolving and removing the chromium deposit with its top layer. Thus a negative form of the desired copy of the original can be obtained. It is claimed that the process is particularly suitable for the production of measuring scales on instruments. The thickness of the films forming the scale images is about 0.1 - 1μ and the reading of such a scale is comparatively easy as it is substantially independent of the angle of incidence of the light falling on the scale.

Sommaire : Description d'une nouvelle méthode pour l'impression d'échelles, graduations, etc. sur verre, pièces d'instruments en métal ou plastic, qui utilise le procédé d'évaporation sous vide.

J. Heidenhain *Brit. Pat.* 696 036

II — Vacuum Apparatus and Auxiliaries — II

Abstract No. and References

20 — PUMPS — 20

Pick a Vacuum System for Your Job 1/II

See Abstract No. : 6/I

A Portable High Vacuum System 2/II

See Abstract No. : 1/I

Procedure for the Graphical Calculation of Multi-Stage Ejector Pumps 3/II

Germany. Single-stage ejectors operated by gas, liquid or steam have a rather inflexible performance. For instance, no change in design will improve the vacuum obtainable by them. If higher vacua are required, multi-stage pumps must be used. In the present article the design and performance of multi-stage steam ejectors is discussed. The methods applied to condense the steam between the stages are of little importance but the temperature at which the condensation is carried out might have considerable effect. The performance of a multi-stage ejector is governed by the dimensions of the individual ejector units and by the requirement of assembling the multi-stage ejectors from individual units of truly complementary performance quality. The essential design features of the individual unit are : The diameter of the diffuser at its narrowest point, the dimensions of the outlet and throat of the actual jet and the ratio of the two latter dimensions. The author examined the relations between these design features and the maximum attainable pumping speed, optimum obtainable vacuum and required steam pressure under certain well-defined conditions. As a result he developed nomograms which contain sufficiently accurate information to give practical guidance in the construction of multi-stage ejectors for a wide range of operational requirements. Two examples of calculating 4–stage and 5–stage ejector pumps on the basis of the nomograms are given. The working costs of various ejector apparatus are shown in a table partly reproduced below.

Operational Costs of Steam Ejectors

Types of Multi-Stage Ejectors	*Technical Data*			*Steam Consumption*			*Water Consumption*			*Total Operational Costs*
	Pumping Speed Kg./hr.	*Vacuum Obtained mm. Hg*	*Steam Pressure Kg./cm²*	*kg./hr.*	*ton/ Year*	*DM/ Year*	*m³/hr.*	*ton/ Year*	*DM/ Year*	*DM/ Year*
a	23	2.0	2.7	580	5,000	40,000	20	175,000	7,000	47,000
b	60	9.0	2.0	670	5,850	47,000	20	175,000	7,000	54,000
c	40	9.0	2.2	755	6,600	52,800	30	263,000	10,500	63,300
d	42	12.0	3.0	370	3,250	26,000	15	130,000	5,200	31,100
e	22	18.0	2.4	235	2,000	16,000	10	87,500	3,500	19,500
Steam Cost; 8 DM/t				Water Cost; 0.04 DM/t			DM = Deutsche Mark			

Sommaire : Les rélations existantes entre certaines charactéristiques de projet et d'éxécution d'éjecteurs à étages multiples ont été étudiées, et certaines données sont presentées sous forme de nomogrammes, facilitant la détermination de toutes les dimensions essentielles d'éjecteurs à étages multiples, requis pour un large domaine d'applications.

Article by L. Zimmermann *Chem. Ing. Tech.* **25**, Nov. 1953 665-671

Oil Diffusion Pump in Glass with a Novel Fractionating Chimney 4/II

Germany. Oil diffusion pumps compared with mercury diffusion pumps have the important advantage that they need no cold traps for their operation in many instances where mercury pumps require a trap, because the oils have a lower vapour pressure than the mercury. However, unless the oil pump is of the fractionating type the vapour pressure of the fuel will rise during service as a result of cracking phenomena in the oil. There are various all-metal types of fractionating oil diffusion pumps available. The boiler of these pumps is divided into a number of annular sections by means of concentrically positioned tubes of varying diameters each leading to jets positioned at different heights in the pump. The liquid oil returned along the wall of the pump casing, on reaching the boiler, moves through all sections before it reaches the centre of the boiler. On its way, its higher vapour pressure components are re-evaporated and returned to the corresponding jets of the pump. Thus, the oil collecting in the innermost section of the boiler has the lowest vapour pressure and is fed exclusively to the high vacuum jet. All-glass fractionating oil diffusion pumps have never become available because a

Vol. 4 195

II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

reproduction of this design in glass is impracticable. In the present article a specially designed all-glass fractionating pump is described which has two jet tubes and provides for efficient fractionating in three stages. It consists of a cylindrical casing with the fore vacuum outlet at the bottom. On the inside of the casing, just below the fore vacuum outlet, is a small annular trough supporting the outer jet tube. The bottom edge of the latter is cut away in several places. The boiler, joined to the lower end of the cylindrical casing, is conical in shape and has a base, wider in diameter than that of the cylindrical pump casing. A portion of the boiler base is projecting downwards. Into this cavity fits the narrow high vacuum jet tube, the bottom edge of which is cut away as in the previous case to let the liquid oil pass. The heater element is wound in two sections. Each section is inserted into glass tubing joined to the boiler base, on the inside. One section heats the oil in the central area for the high vacuum jet and the other section heats the oil in the area outside the high vacuum jet tube. In operation, the fuel returning from the jets collects in the trough from where its most volatile fractions are removed through the fore vacuum outlet. The remainder penetrates into the boiler, reaching the outer area first. Here, the fractions of intermediate vapour pressures are re-evaporated. Only those of the lowest vapour pressure reach the central area. For correct operation, the power dissipated in the two heater sections must be controlled to give the appropriate differential temperatures. When the pump was operated with Leybold Oil ' F ', it had a speed of 70 l./sec. and gave an ultimate vacuum of 5×10^{-6}mm. Hg at 0.1mm. Hg backing pressure. Leybold Oil ' F ' has a viscosity of 198 cS. at 20°C. After 12 hours use in the pump it was split into three fractions of 330 cS., 183 cS. and 91.6 cS.

Article by W. Frank *Glas-und Hochvakuum Tech.* **2**, Dec. 1953 294-296

O *Sommaire* : Description d'une pomp fractionnante à diffusion à huile, entièrement fabriquée en verre, donnant un vide maximum de 10^{-6}mm. Hg avec un vide préliminaire de 0.1mm. Hg.

Improvements i.o.r.t. Vapour Vacuum Pumps 5/II

United Kingdom. The fluid of a diffusion pump is a liquid which is vaporised during operation in the boiler of the pump. As a result of the heating the surface of the liquid in the boiler is agitated, producing considerable splash and spray. In ordinary diffusion pumps drops of liquid may be carried upwards by the rising vapours until they reach the vapour jets. Their presence on or near the jets is likely to have serious adverse effects on the efficiency of the pump such as the following :—Part of the kinetic energy of the vapour stream through the jets is wasted because it is absorbed by the transfer of droplets from the jet to the cooled wall of the pump. This transfer does not contribute to the pumping action, but merely constitutes a waste of the heat put into the boiler. Some droplets may evaporate from positions on the wall of the jet support and these vapour molecules may diffuse in a direction opposed to the pumping action resulting in reduced pumping action and back-streaming of vapour molecules into the vessel pumped. The present invention aims at eliminating these adverse effects by providing a combination of baffles which intercept and change the direction of the fluid droplets rising with the vapours from the boiler. Various systems of baffles have been designed to suit both single- and multi-stage diffusion pumps. In the case of a two-stage pump, described in detail, the system consists of a annular shaped baffle with a large hole, joined at its periphery to the inside wall of the central vapour tube, and a circular disc, slightly larger than the hole in the annular baffle fixed to the central jet-supporting rod in a position somewhat below the annular baffle. The distance between both baffles is such that between them they offer an effective barrier to the rising drops of liquid. By making provisions for the adjustment of the distance between the two baffle members a throttling effect can be obtained which facilitates ' drying ' the vapour stream to varying degrees.

B. D. Power & Edwards High Vacuum Ltd. *Brit. Pat.* 700,978

Sommaire : Description d'un baffle inséré dans la tuyère d'injection de pompes à diffusion, empêchant les gouttes du fluide de la pompe de remonter vers les éjecteurs.

O **A Metal High Vacuum Manifold** 6/II

See Abstract No. : 26/I

21 — GAUGES — 21

Techniques for Measuring and Controlling Vacuum 7/II

See Abstract No. : 3/I

Wide-Range Vacuum Gauge 8/II

United States. An improved design of alphatron-type gauges is described. The gauge can be used for pressure measurements from 1,000 to 0.0001 mm. Hg. This instrument has six ranges. Indication is linear. This has been made possible by eliminating recombination of ions produced by the alpha particles (positively charged helium atoms emanating from the radium source) at higher pressures through the provision of two separate ionisation chambers of different size. The smaller chamber is used for the three high pressure ranges of 1,000, 100 and 10 mm. Hg. Contamination of the electrodes is avoided by using a low-voltage collection field. The size of the head of the new gauge is reduced to a metal cylinder of about 50 c.c. volume as compared with 450 c.c. in the conventional design. The metal envelope contains the radium source, the ionisation chambers, the collector electrodes, pre-amplifier, thermal relays and range resistors. Details of the circuitry are discussed and shown in an illustration. Basically, the circuit is that developed by Moody in 1939. A CK-571 AX electrometer

II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

pentode is used in the input stage and a CK-546 DX pentode in the cathode follower. There are three thermal relays. Two connect the high and low pressure chamber respectively to the electrometer grid. Closing both relays at the same time, the condition of zero pressure in the chamber is simulated and this facilitates the zero setting of the instrument. The third relay facilitates the changing of the input resistance to the amplifier. For practical reasons the value of the input resistance is limited to 100,000 MΩ. This determines the lower limit of the measuring range of the instrument as given above. At the same time this resistance value determines the input time constant at about 5 seconds but this value is further reduced by special provisions so that the actual response of the meter is less than 1 second.

Sommaire : Des détails sont données sur une gauge à ionisation du type alphatron, ayant 6 gammes de mesure de 1,000 à 0.0001 mm. Hg, et montée avec deux chambres d'ionisation pour éliminer la recombinaison des ions.

Article by C. B. Sibley & J. R. Roehrig *Electronics* **26**, Nov. 1953 176-177

A Differential Capacitance Manometer

9/II

United Kingdom. The capacitance manometer described in this article represents an improved design of the type of instrument employed to record pressures in the range 0.03–15 mm. Hg developed by a miniature Pitot head of a blood flowmeter. In the previous design the diaphragm, on which the pressure difference acts, forms an integral part of the capacitance pick-up. The pressure is transmitted from the Pitot head by means of a saline solution. The sensitivity of the previous design was poor because the modulus of volume elasticity of the diaphragm was too large with respect to the pressures applied and, once the inter-electrode gap had been made a minimum, no further corrective action was possible. Consequently trouble was experienced with drift resulting from low overall signal-to-noise ratio, when a highly sensitive circuit was used. The new design has a relative sensitivity, 25 times that of the previous instruments. Its essential new feature is the separation of the pick-up unit from the diaphragm in contact with the filling solution. The two components are linked by a lever mechanism, the counterbalanced lever rod being retained in a (half) cylindrical membrane serving as a fulcrum. The length of the lever from fulcrum to end is 7.6 mm., its ratio being 1 : 1. The diaphragm is top-hat shaped, the brim serving as the mounting flange. A stainless steel wire soldered to the centre of the diaphragm connects with one end of the lever. The diaphragm has a diameter of 4 mm. and is $\sim 25\mu$ thick. Its modulus of volume elasticity is $\sim 4 \times 10^9$ dyn/cm^5. The membrane is $\sim 40\mu$ thick and has a diameter (projected aspect) of 6.4 mm. The other end of the lever carries the moving electrode in the form of an aluminium disc. The fixed electrode is a flat plate and mounted on (but insulated from) a rigid support. A screwed covering cap is provided which houses a small inductance, resonating with the pick-up capacitance. The effective diameter of the capacitance pick-up is 12.7 mm., the gap $\sim 30\mu$ and the total capacitance 43 pF. Special arrangements for damping are made in order to make the response of the instrument nearly dead-beat.

Sommaire : Description d'un nouveau manomètre à variation de capacité du type employé dans les mesures de pression du sang, et courant une gamme de 0.03 à 15 mm. Hg.

Article by I. G. Baxter *J. Sci. Instrum* **30**, Oct. 1953 358-360

Improvements i.o.r.t. Apparatus for Measuring Gas Pressure

10/II

United Kingdom. It has been found that the conventional Pirani-type gauge requires frequent recalibration. It is believed that the changes in the performance of the gauge which necessitate recalibration are due to changes at the surface of the filaments resulting from chemical attack by gases, the pressure of which was measured, *e.g.* the formation of oxides or nitrides in the case of air. Normally the material of Pirani gauge filaments is tungsten. To give better protection against chemical attack it is proposed in this patent to coat the tungsten filaments with a material of the desired property which at the same time should possess electrical characteristics, similar to those of tungsten. A suitable material is rhodium. In one application the coat of rhodium, approximately 1μ thick, is applied to filament coils of 190 turns, stretched to a length of about 72 mm., and made from tungsten wire 44μ diameter, 500 mm. long. The filaments are first cleaned by immersion in caustic soda solution followed, after a water rinse, by swilling in a dilute sulphuric acid solution which forms part of a deplating bath and subsequent etching. The rhodium is then electrodeposited on to the filaments in a bath containing dilute sulphuric acid and a suitable rhodium plating solution. In the process the filaments form the cathode and the anode is of platinum. The deposition is carried out at a rate of 2 mg./A.min.

Sommaire : Proposition est faite de recouvrir les filaments de tungstène de gauges de type Pirani avec du rhodium comme protection contre les attaques chimiques de gaz, lesquels étant mesurés dans ces gauges.

General Electric Co. Ltd. & J. D. Buckingham *Brit. Pat.* 699,527

Repairing Ionisation Gauge Tubes

11/II

United States. This note describes a method of replacing a broken or burnt out tungsten filament in the VG–1A type ionisation gauge. The glass tube is cut about 1 inch from the bottom by the hot wire method, applying heat for 45-60 seconds. The old filament is removed and a new hairpin filament of 0.008 inch pure tungsten wire is spot welded in position. While open, the interior of the glass tube is rinsed with dilute ammonia, rubbed with a pipe cleaner and the wire supports and grid are cleaned with a sodium hydroxide solution followed by an ammonia rinse and distilled water wash throughout. The 2 parts of the glass tube are joined together, after exactly relocating the parts in relation to each other, using a medium-size flame from an oxygen gas torch, clamped to the bench. When the tube is cold, it is given a final rinse with distilled water. After thorough outgassing, 3 tubes repaired by this method gave the same pressure reading (within 0.3 in the 10^{-6} mm. Hg range) as a new tube.

Sommaire : Description d'une methode simple de remplacement des filaments brûlés dans des gauges a ionisation VG–1A.

Note by A. J. Stinnett *Rev. Sci. Instrum.* **24**, Oct. 1953 985-986

Vol. 4 1954

II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

22 — MEASURING PLANT (VACUUM) — 22

A Mass Spectrometer for Leak Detection 12/II

See Abstract No. : 33/II

A Metal Vacuum Lock for a Mass Spectrometer 13/II

United States. A vacuum lock is described which was developed for use with an all-metal mass spectrometer employed for routine isotopic determination of elements by solid analysis. Without a lock, a change of sample required breaking the vacuum in the source chamber and pumping down from atmosphere after insertion of the new sample. While the actual change can be made in about 5 minutes, about 90 minutes are required to attain the operating pressure of approximately 1×10^{-6} mm. Hg. Each analysis takes 20 minutes. By providing the vacuum lock described in this report the pressure in the source chamber never rises above 7×10^{-6} mm. Hg during an exchange of samples. Consequently pumping-down time is reduced to approximately 25 minutes. The lock consists of a small chamber connected to the source chamber by a gate valve operated by an external lever. The lock is evacuated by a separate mechanical pump. The sample is placed in a small retort suspended from a rod. The rod with the sample is inserted in the lock. After pumping down the lock, the gate valve is opened and the retort is pushed into the furnace of the ion source by means of the rod. The removal of the spent sample is carried out accordingly. While the gate valve is open the vacuum pump serving the lock is shut off by a special valve.

Report by C. R. Fultz L. V. Wilson & Carbide & Carbon Chem. Corp. *Instrumentation Report* No. Y-990 July, 1953

Sommaire : Un sas à vide, utilisé dans un spectromètre de masse employé pour l'analyse de solides, est décrit. Son emploi réduit le temps de pompage de 90 à 25 minutes, après changement des échantillons.

Mass-Spectrometric Determination of Gas Impurities Separable by Condensation 14/II

See Abstract No. : 2/IV

A Rotating Anode Gas X-Ray Tube 15/II

United Kingdom. As a result of certain modifications of the conventional Mueller-Shearer X-ray tube the cathode beam could be focussed so sharply on the target that the water cooling system was no longer effective enough to prevent the target surfaces from being punctured. The cathode beam had to be defocused with a consequent reduction in efficiency. This difficulty has been overcome without altering either the water cooling or the vacuum pumping system by providing a rotating anode. Details of the new design are given. The target, a short length of a hollow metal cylinder of $1\frac{1}{8}$ inch diameter, is fitted to a vacuum-sealed hollow shaft and rotated at about 320 r.p.m., driven through gears by a small a.c. motor. The target is situated in a chamber connected directly to the X-ray tube. The cathode consists of an aluminium rod sheathed in a brass tube which projects about 2 mm. beyond the end of the rod in order to give a sharp focus of the beam. The target is cooled by circulating water down a central stationary tube situated inside the hollow shaft and returning it through the space between the central tube and the wall of the hollow shaft. Demountable connections in suitable positions enable the targets to be exchanged in about 10-15 minutes. The rotating anode makes it possible to obtain a finer focus and a focal spot of higher brightness than could be obtained by alterations to the water cooling system. The new tube is steadier in operation than the tube with a stationary anode and may be left running for hours without any adjustment of the vacuum system becoming necessary.

Article by E. A. Owens *J. Sci. Instrum.* 30, Nov., 1953 393

Sommaire : Des détails sont donnés sur une modification apportée au tube à rayons –X de Mueller-Shearer, qui remplace l'anode fixé et permet d'obtenir un rayon bien concentré sans effet préjudiciable sur la cible.

The Diffraction of 150 kV Electrons 16/II

United Kingdom. It was established in earlier experiments employing accelerating voltages ranging from 800 V to 75 kV that, in the case of transmission, the higher the voltage, the higher was the ratio of elastically to inelastically scattered electrons and this resulted in better pattern clarity and freedom from background. Similarly, in the case of reflection from poorly conducting surfaces, interference from charging up of the specimen was reduced and permitted the lowering of the minimum grazing angle between beam and specimen surface. Experiments carried out by Moellenstedt using the transmission technique indicated that a further increase of the accelerating voltage beyond 75 kV might be beneficial. The authors carried out experiments increasing the voltage to 200 kV. A three-electrode hot-filament source was used. The camera embodied a two-lens focusing system, both lenses being iron shrouded and situated outside the vacuum. The image of the hot-filament source was brought to a focus a short distance below the upper lens, reducing the image size of the source about 10 times. The second lens focused the beam on the screen without further reduction. Details of the arrangement are given. A mica single crystal, about 5,000 Å thick, examined by the transmission technique at 60, 100 and 140 kV with the beam parallel to the C–axis of the crystal gave patterns which were the clearer, the higher the electron speed. A corresponding effect was obtained, using the reflection technique, when a rock salt single crystal was examined at 60 kV and 120 kV alternatively. The angle of incidence used in both cases was 1°. In order to examine the effective depth of penetration at low grazing angles, the mechanically

II — Vacuum Apparatus and Auxiliaries — II

Contd.

Abstract No. and References

polished surface of a crystal of quartz was examined by reflection at 1° angle of incidence and 60 kV. A faint veiling of the pattern near the shadow edge, apparent in this experiment, could be greatly reduced by increasing the voltage to 140 kV even when the angle of incidence was reduced to 10 minutes. But at this low angle of incidence diffuse haloes became visible. Examining a fresh cleavage of muscovite mica under exactly the same conditions no such haloes could be observed and this proved that the haloes in the case of the quartz crystal were due to the presence of an extremely thin polish layer of amorphous quartz. As the pattern of the surface of the quartz crystal was still visible it was possible to determine the thickness of the polish layer from the angle of incidence (10 minutes) and the angle of emergence (1° and 50 minutes). In this particular case the thickness of the amorphous layer was 5 Å. In a similar manner the thickness of the amorphous polish layer on a corundum crystal could be determined to be of the order of 1 or 2 atom layers. The experiments showed that the use of accelerating voltages in the region of 140 to 150 kV permits a wider range of specimen thicknesses in the transmission technique and in the case of the reflection technique the restriction of the beam penetration to a depth just beyond the top atom layer, as long as atomically smooth non-conducting surfaces are examined.

Sommaire : Description est donnée d'une caméra à diffraction d'electrons, opérant sous des tensions allant jusqu' à 150 kV. Les avantages de l'emploi de hauts potentiels d'accélération sont discutés et expliqués.

Article by G. I. Finch H. C. Lewis & D. P. D. Webb *Proc. Phys. Soc.* **66B**, Nov., 1953 949-953

An Improved Electron Diffraction Instrument

17/II

United States. The design of the original General Electric electron diffraction camera is reviewed in the light of practical experience with the instrument. Much attention has been given to a change of the original optical system which consists of the electron gun with an apertured cathode, an anode with two alternative apertures, a condensing lens and a collimating aperture before the specimen. The insertion of lenses after the specimen, for instance, has been considered but did not prove adequate for contrast improvement. The designers feel that electron diffraction instruments for the purpose of examining fine structure in minute specimens may warrant multi-lens systems but should be combined with electron microscopes. The ' independent ' electron diffraction camera should have as simple an optical system as possible. However, the use of velocity filters has great advantages as they make it possible to photograph strong patterns which were obscure or invisible in the past and provision has been made for their inclusion. These filters remove from the image inelastically scattered electrons, which have only lost an insignificant amount of their energy in the process and, therefore, only add to the general background. Other modifications or additions are : A shadow microscope attachment, a new specimen-to-plate measuring device built into the specimen chamber, O–ring and grommet seals replacing the flat gaskets and Wilson seals previously used, and a change in the construction of the camera chamber. In the past the chamber was fabricated from plates and the joints were brazed. The pressure exerted on certain parts of the housing, when evacuated, is nearly 1 ton. This resulted in cold working of the joints and warping of the plates. The new chamber is cast in one piece from aluminium. The thickness of the walls is strengthened and certain sections are curved.

Sommaire : Les parties essentielles du modèle original de la caméra à diffraction d'electrons de G.E., sont examinées et critique en est faite; les modifications apportées durant ces dernières années sont discutées.

Paper by M. J. Columbe & S. P. Newberry *Electron Microscope Soc. Am.* Nov., 1953

A ' Double ' Camera for Electron Diffraction

18/II

Japan. It is the normal practice in electron diffraction measurement to observe the material to be examined and the reference material separately in the instrument. This procedure involves the risk that the wavelength of the electrons may have changed between the observations. The author reports a new method, whereby the two diffraction patterns are observed simultaneously using a ' double ' camera. The two units have a common h.t. supply and consequently the accelerating potential in both units is the same. Two practical examples are given. In the first, two pieces of gold foil are examined simultaneously in two units with a camera length of $L_1 = 150$ mm. and $L_2 = 227$ mm. respectively. The ratio of the rings observed in the first (D) and second apparatus (D^1) is $D^1/D = 1.518$. This compares with the ratio $L_2/L_1 = 1.51$ and proves that the energy of the electrons was the same in both instruments. This ratio is called the camera constant, C. In the second example, gold foil and magnesium oxide smoke are examined simultaneously in the same combination. With the help of the camera constant the spacings of the magnesium oxide crystal (d_2) can be calculated from the formula : $(d_2 \times D_2) / (d_1 \times D_1) = C$. The values obtained for d_2 are in agreement with the spacings of magnesium oxide crystal determined by X-ray diffraction. The ' double ' camera is most useful where the variation of accelerating voltage is greater than 670 V in 5×10^4 V.

Sommaire : L'emploi de deux caméras, alimentées par une source de haute tension commune, dans la diffraction d'electrons, facilite l'observations simultanée du materiau examiné et du materiau de référence.

Article by S. Yamaguchi *J. Appl. Phys.* **24**, Oct., 1953 1305-1306

The Behaviour of Light-Sensitive Evaporated Layers in the Electron Microscope

19/II

Germany. A thin silver film, evaporated on collodion is examined before and after conversion to silver bromide and then during irradiation in the electron microscope. The initial uniform distribution of single crystals of silver is converted to well separated (200 mμ) droplets of silver bromide which after irradiation have the appearance of having exploded. The unit cell dimensions of the ' bromide ' layer are intermediate between those of silver and those of silver bromide. The reason for this is still doubtful.

(*Science Abstracts*)

Sommaire : Le comportement d'un film d'argent évaporé avant et après sa conversion en bromure d'argent pendant l'exposition dans le microscope électronique, a été étudié.

Note by H. Haardick *Naturwiss.* **40**, No. 14, 1953 386

II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

Fine Structure in Polyethylene Terephthalate Fibres 20/II

See Abstract No. : 23/II

26 — MATERIALS — 26

High Temperature Materials for Vacuum Service 21/II

United States. Materials used in evacuated apparatus or plant have to meet the following essential requirements, Low vapour pressure, stability in low-pressure atmospheres, ease of outgassing and low gas permeability. In addition, there are certain materials which are of importance for the reason of their gettering properties. This article deals with the construction materials of two major classes of components and plants, *i.e.* thermionic valves and vacuum furnaces. Valve materials are subjected to temperatures ranging from 400 to 4,100°F and operate in vacua of the order of 10^{-8} mm. Hg. Anodes for low-power receiving tubes, or valves, are being manufactured to an increasing extent from aluminised steel which is readily fabricated but less costly than nickel. Where the anode is run at high temperatures, graphite, molybdenum and tantalum are used. The materials for grids vary from nickel and manganese alloys to molybdenum or molybdenum alloys. Molybdenum grids usually employed in high-power tubes are cladded with zinc, platinum, gold or silver, in order to restrict electron emission. The most important material for electron emitters is tungsten or thoriated tungsten normally employed in the form of filaments. The filaments or indirectly heated cathodes of low-power tubes can be made of pure nickel, a nickel alloy or tungsten, coated with a mixture of barium, strontium and calcium oxides. It follows a description of the use of getters. The operating characteristics of bulk and powder-type getters are shown in a table, reproduced below :

Properties of Bulk and Coating Getters

Material	*Form of Getter*	*Outgassing Temp. °F*	*Operating Temp. °F*
Zirconium	Sheet, wire or powder	1,300–2,350	1,500
Tantalum	Sheet, powder	2,900–3,600	1,300–2,200
Thorium	Powder	on metal base, 1,500–1,800 on graphite base, 2,700–2,900	750–925
Thorium-Mischmetal	Powder	1,475–2,200	400–900
Columbium	Pellets	3,000	900

The problem of suitable materials for vacuum furnaces is much less explored than that of materials for thermionic valves. There are induction, resistance, arc and gas-fired vacuum furnaces. The first group is employed for the treatment of high melting point metals. Small melts may be carried out in crucibles made of alumina, magnesia, beryllia, silimanite, quartz, thoria, zircon, stabilised zirconia or graphite. Where carbon pick-up by the melt must be avoided, graphite crucibles are sometimes lined with a ceramic liner. The stability of the refractories sets the maximum temperature at about 3,200°F, for which commercial induction furnaces are available. The properties of a number of refractories are shown in a table reproduced on the next page. For treatment at temperatures not exceeding 2,500°F externally-heated resistance furnaces can be used. But in small resistance-heated furnaces fitted with tungsten or molybdenum heaters inside the vacuum chamber extremely high temperatures can be produced. Ceramic tube and muffle furnaces are also referred to. Vacuum arc furnaces for melting metals such as titanium and zirconium which react readily with most materials and for that reason cannot be melted in crucibles, are mentioned. The same type of furnace is used for the production of molybdenum because of its extremely high melting point. The vacuum thermal reduction of metallic compounds and other comparatively low-temperature operations are carried out in gas-fired evacuated retorts consisting of thick steel alloy tubes. Besides the tables reproduced here the article carries two graphs and four tables on the vapour pressures of various high melting metals, the dissociation pressures of various oxides, the properties of typical metals and alloys and the general characteristics of construction materials suitable for plant operating below and above 2,200°F.

Sommaire : Une étude détaillée et étendue sur les caractéristiques de materiaux employés pour la fabrication de pièces et appareils opérant sous des pressions basses et des températures élevées.

Article by H. B. Michaelson *Mater. & Meth.* Dec., 1953 110-115

II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

Table to Abstract 21/II

Properties of Non-Metallic Materials

(All properties depend on impurities and fabrication method)

Material	*Melting Point, °F*	*Max. Operating Temp. in High Vacuum, °F*	*Coef. of Ther. Exp. Per °F*	*Thermal Cond. Btu/Hr/Sq Ft/Ft/°F*	*Thermal Shock Resistance*	*Hardness Mohs' Scale*	*Vapour Pressure*		*Elect Resistivity at 2,200°F, ohm-cm*	*Load Bearing at 2,200°F*
							mm. of Hg	*Temp F*		
Alumina, fused	3,722	3,270	4.5×10^{-6} (68–2,300F)	0.4–0.5	Excellent	9	0.35	3,720	10^{4}	Good
Beryllia, fused	4,568	3,630	$5.1–5.2\times10^{-6}$ (68–2,550F)	1.1–2.1	Excellent	9	1.8×10^{-3}	3,775	10^{13}	Good
Graphite	—	4,530	4.3×10^{-6} (77–104F)	4.4–14.4	Excellent	0.5–1.0	10^{-6}	3,630	7.9×10^{-4}	Excellent
Magnesia	5,072	2,910	$7.9–8.3\times10^{-6}$ (68–3,100F)	0.4–0.8	Poor	6	High	>3,630	5×10^{-11}	Fused, good; sintered, poor
Mullite	3,250-3,335	2,730–3,090	$2.5–2.9\times10^{-6}$ (68-2,600F)	0.2–0.3	Good	6	—	—	—	Excellent
Silica, fused	3,110	2,010	0.3×10^{-6} (68–2,280F)	0.2–0.35	Excellent	5–7	9.9×10^{-6}	1,830	5×10^{5}	Fair
Thoria, fused	5,486	4,900	$5.2–5.3\times10^{-6}$ (68–2,550F)	Very low	Poor	7	3×10^{-2}	4,710	10^{10}	—
Zircon	4,622	3,090	$2.0–3.7\times10^{-6}$ (68–1475F)	0.2–0.3	Good	8	—	—	10^{4}	Good
Zirconia, stabilised	4,600-4,700	3,990	$2.8–6.0\times10^{-6}$ (68–2,275F)	0.36	Fair to good	8.5	—	—	—	Good
Silicon Carbide	>4,900	3,990	$2.5–2.6\times10^{-6}$ (32–3,090F)	1.1–2.4	Good	9–10	—	—	2.5	Excellent
Lava, fired	—	2,190–2,280	$5.8–6.4\times10^{-6}$ (77–2,900F)	—	—	6	—	—	10^{4}	Good

Use of P.T.F.E. In Impregnating Porous Materials

22/II

United Kingdom. In connection with work necessitating the electrolysis of molten sodium hydroxide/potassium hydroxide mixtures no metals could be found suitable for use as anode material. Even graphite crucibles released appreciable amounts of iron into the melt with the attendant danger of contaminating the cathode. The difficulty was eventually overcome by impregnating the graphite under vacuum with a dispersion of Polytetrafluorethylene (Teflon). The amount of iron released from anodes, treated in this manner was greatly reduced. At the same time the anodes were not attacked by the molten mixtures even at temperatures near 250°C. It is suggested that P.T.F.E. may be suitable for the impregnation of other porous materials subject to chemical attack.

Letter by H. Hughes *Chem. & Industr.* 12.12., 1953 1342

Sommaire : Après une étude poussée sur les matériaux appropriés pour la fabrication d'anode, on a trouvé que les anodes, faites en graphite imprégne de Teflon sous vide, donnent des résultats satisfaisants dans l'electrolyse de mélanges de NaOH/KOH fondus.

Fine Structure in Polyethylene Terephthalate Fibres

23/II

United Kingdom. Kassenbeck demonstrated that drawn polyethylene terephthalate fibres possessed a surface skin about 2,500 Å thick by making and photographing in the electron microscope polystyrene-silica replicas of specially prepared sections. He also showed that beneath this skin lies a fine structure of oriented fibrils which appear to vary in diameter between 250 Å and 750 Å. The authors have extended these investigations by tearing open the monofilament along the axis and making germanium replicas of the exposed surface by the vacuum evaporation technique. The latter technique is simpler than the polystyrene-silica method. In addition, germanium yields a completely structure-free replica and does not tend to crystallise in the electron beam. The results suggest that there are ' fundamental ' fibrils of about 200 Å diameter and that some of these group together resulting in fibrils of up to about 1,000 Å diameter.

Letter by A. Cobbold R. de P. Daubeny K. Deutsch & P. Markey *Nature* **172**, 31.10., 1953 806

Sommaire : La structure des fibres de polyethylene terephthalate passées à la filière a été examinée avec l'aide de repliques de germanium obtenues par la technique de l'évaporation sous vide.

Density of Deposited Carbon

24/II

United Kingdom. A method is described of depositing elemental carbon from gaseous compounds containing carbon, such as methane by passing the gas in a closed vessel over a heated graphite rod of 5 mm. diameter at an initial pressure of 15 cm. Hg. The coating obtained was 1–2 mm. thick and its maximum density was found to be 2.22 g./cm³ (the theoretical lattice density is 2,265 g./cm³). Varying the temperatures of the graphite rods and the initial pressures of the methane gas it was found that the density of the deposits rose with the temperature of the rods and that, at constant temperature, the density of the deposit rose with the initial

II — Vacuum Apparatus and Auxiliaries — II

Contd.

Abstract No. and References

pressure of the gas. The structure of the deposits was examined by X-rays and by normal metallographic techniques. By the former technique it was established that the high-density deposits consisted of sheets of graphitic carbon the c–axes of which were normal to the surface of the rod. Low-density deposits showed a similar structure but less pronounced alignment. The electrical resistivity of high-density deposits at room temperature, measured in the direction parallel to the axis of the rod, was 35×10^{-5} ohm cm. Generally, the resistance depends on the density. The variations in the properties of carbon deposited from hydrocarbons at high temperatures and low pressures, as established in these experiments, should be taken into account, where such deposits are used for the study of chemical phenomena such as the kinetics of the oxidation of carbon.

Sommaire : Une étude a été faite sur les propriétés de carbone deposé sous basses pressions provenant de gaz d'hydrocarbures et à hautes températures.

Letter by A. R. G. Brown A. R. Hall & W. Watt *Nature* **172**, 19.12.1953 1145-1146

Rate of Evaporation of Glycerol in High Vacuum 25/II

See Abstract No. : 39/I

28 — APPARATUS COMPONENTS — 28

A Metal High Vacuum Manifold 26/II

See Abstract No. : 26/I

Copper Isolation Trap for Vacuum System 27/II

United States. Traps cooled with liquid nitrogen have the following shortcomings : (1) When the level of the refrigerant is lowered the condensed gases re-evaporate. (2) The molecules of condensible impurities contained in the permanent gas, while at comparatively high pressures, may be stopped from diffusing to the cooled wall and may pass through the trap. The new trap effectively overcomes these difficulties and is simple, inexpensive and effective even at room temperature. It consists of a conventional glass tubular re-entrant trap with a strip of corrugated copper foil, formed from OFHC copper sheet 0.003 inch thick and 6 inch. wide, wound layer upon layer around the central vertical tube inside the outer tube. The gas and the entrained condensible vapours pass down the central tube and up through the small diameter copper ' straws ', formed by the superimposed corrugations, to the outlet of the trap. The corrugations are about 0.025 inch. deep and thus form individual passages of about 0.050 inch. diameter. When used with refrigerants the large surface area and high thermal conductivity of the trapping surfaces ensure that the copper foil remains cool although the refrigerant level may vary. After baking the whole system including the trap it was found that the trap was just as effective whether or not the trap was cooled with liquid nitrogen. Traps of this kind have proved to be highly effective in preventing backstreaming of oil vapours for oil diffusion pumps, even at room temperature. In one case, due to its presence, a pressure of 10^{-9} mm. Hg could be maintained in the system for periods of a week or more.

Sommaire : Le modèle amélioré d'un piege à refroidissement qui est également éfficace avec ou sans refrigérant est décrit.

Note by D. Alpert *Rev. Sci. Instrum.* **24**, Oct., 1953 1004

Metal Vacuum Lock for a Mass Spectrometer 28/II

See Abstract No. : 13/II

A Rotary Vacuum Seal 29/II

United Kingdom. A widely used type of shaft seal which is normally used in water pumps and oil refining equipment has been tested for its use as a rotary vacuum seal. A fixed flat compounded carbon ring is sealed to the body of the unit which is fitted to the vacuum chamber. The shaft, which passes freely through the carbon ring is vacuum sealed to a flat stainless steel ring positioned on the inside of the vacuum chamber. These two rings are kept in firm contact by a helical spring arranged so that the stainless steel ring turns with the shaft. The space between the shaft and the seal housing is filled with oil to increase the efficiency of the seals and lubricate the sliding surfaces. A 1-inch. diameter shaft sealed with Apiezon ' B ' oil and rotated at 600–900 r.p.m. was fitted to a $1\frac{1}{2}$ litre vacuum chamber evacuated by an oil diffusion pump unit. A pressure of 2×10^{-4} mm. Hg was maintained in the chamber for a period of 220 hours and 2×10^{-5} mm. Hg was attained for short periods. It is suggested that the efficiency of the seal could be improved by reversing the position of the rings and the spring so that the atmosphere could act on the oil in the seal and aid contact between the sliding surfaces.

Sommaire : Un joint pour arbre de transmission normalement employé dans les machines hydrauliques a été essayé avec de bons résultats comme joint dans les parties tournantes sous vide.

J. Sikorski & H. J. Woods *J. Sci. Instrum.* 30, Nov., 1953 432

II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

Improvements r.t. the Sealing of Joints in High Vacuum and the Like Systems

30/II

United Kingdom. This invention relates to sealing rings of the type inserted between the flanges of two pipe sections in order to effect a vacuum-tight seal. The sealing ring consists of a toroidal rubber ring retained in a pair of metal rings, one being larger in diameter than the rubber ring and the other, smaller. The present design represents an improvement on a similar arrangement described in British Patent No. 656,032. The inner and the outer metal ring are grooved on the surface facing the rubber ring. The grooves are V–shaped, the V being of an angle of about 140° and the surfaces of the V are positioned at an angle (θ) of about 20° against the vertical. In order to counteract any out-of-parallel position of the flange surfaces of the pipes and the consequent danger of distortion during clamping, the cross section of the metal retaining rings are slightly tapered toward the edges of the rings away from the surface facing the rubber ring. The rubber ring has a diameter which is slightly larger than the thickness of the metal rings and its compressibility is assumed to be very low. On assembly, therefore, the rubber ring will be deformed and the grooves should be dimensioned in such a manner that the surfaces of the rubber ring, in that condition, is in contact with all points of the corresponding adjacent flanges and groove surfaces. The correct relationship between rubber ring area and space enclosed by the V–grooves can be determined by calculation using the formulae :

$$\pi r^2 = 2 x y - \tfrac{1}{2} y^2 \tan \theta$$
$$x = r \operatorname{cosec} \theta$$

Where r is the radius of the sectional area of the rubber ring, x half the distance between the apexes of the two V–grooves, and y the maximum thickness of the metal rings.

Sommaire : Description est donnée d'un joint constitué d'un anneau de caoutchouc placé entre deux anneaux métalliques.

G. Burrows & Metropolitan-Vickers Electrical Co Ltd. *Brit. Pat.* 701,614

Modified Hoke Connector

31/II

United States. The standard Hoke cone-type connector may be modified to facilitate its operation and greatly reduce the maintenance required. The method of adapting the connector to use a Neoprene O–ring and the favourable experience with several modified connectors are described.

(*Nuclear Science Abstracts*)

Sommaire : Une modification apportée au point, de type conique Standard Hoke joint qui en reduit l'entretrien est décrite.

Report by E. C. Evans & A. Langdon *A.E.C. Report* Sept., 1953

29 — MISCELLANEOUS APPARATUS — 29

Experience Gained with Various Leak Detecting Methods and Apparatus

32/II

See Abstract No. : 5/I

A Mass Spectrometer for Leak Detection

33/II

France. Leak Detectors which require pure helium for their operation are expensive to use, particularly in Europe. The authors developed a mass spectrometer leak detector which facilitates the use of a gas mixture with a relatively low helium content as the test gas without sacrificing the known sensitivity of that instrument. The work was carried out in two stages : The first was concerned with the design of an instrument with two spectrometer cells operating in cascade formation in order to reduce as much as possible ' background interference '. The second stage was concerned with the construction of an especially efficient ion source. Details of the circuitry associated with the new apparatus are given in Abstract No. 23/I.

Sommaire : On décrit un spectromètre de masse conçu pour fonctionner avec un mélange à faible teneur en helium. Il est caractérisé par une valeur élevée du rapport signal-bruit, obtenue par un montage en monochromateur double et par une source d'ions à rendement élevé dont on décrit l'optique.

Article by C. Cassignol R. Geller & J. Moreau *Le Vide* 8, Nov., 1953 1415-1421

A Few Remarks on the Electronic Circuit of a Leak Detector

34/II

See Abstract No. : 23/I

The Liquid Helium Plant of the Physical Laboratory of the University of Louvain

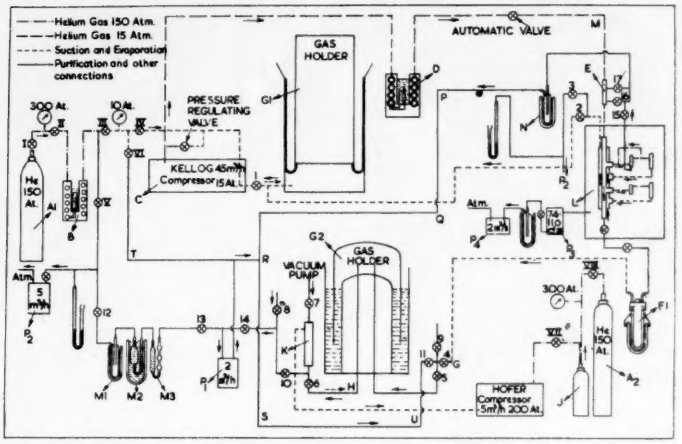
35/II

Belgium. The normal Collins helium liquefier is of the open-circuit type. Its operation entails a substantial loss of helium during each run. As it is difficult to obtain adequate helium supplies in Europe the authors modified such a liquefier, made available to them by the Marshal Aid Plan, by altering it into a closed-circuit type, thus reducing the loss of helium gas to a few litres in each run. The article describes the final construction in all details and includes a diagram of the apparatus shown on the following page :

Vol 4 195

II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References



In order to convert the liquefier into a closed-circuit type the following components have been added :—Needle valves Pos. I–VIII, steam valves Pos. 1–11, stopcocks Pos. 12–14 and all elements belonging to, and including, gas holder G2 which forms the reservoir for the gas saved prior to its recompression and filling into the bottle A_2. The valves 1, 2, 3, facilitate the tracing of leaks in the cryostat, the compressor C and in the respective pipe lines. In operation, gaseous helium is admitted to the system from A1 and reaches the gas holder G1 *via* a charcoal purifier B. From G1 it passes into the cryostat through valve 2. The helium compressed in C flows through the drier D, oil separator and glass wool filter E to the high pressure side of the cryostat. The drier D is operated with liquid air and has been inserted to protect the cylinders of the expansion device. It consumes about 10 litres of liquid air during a 5–hour run. The vapours escaping during siphoning into vessel F1 are transmitted to the second gas holder G2 by the route G–4–5. This holder has a capacity of 1 cubic metre and is filled with transformer oil. The helium collected in G2 is compressed in the Hofer compressor and pumped *via* the oil separator J, filled with silica gel, into the cylinder A_2 to a pressure of 150 atmospheres completing the closed circuit. M1 is a liquid air drier, M2 a charcoal cleaner and M3 a glass wool filter. All three are made of glass. Immediately after completion of a run the purifier B, still at liquid air temperature, is pumped out and the helium liberated in the process is transmitted *via* M1, M2 and M3 to the holder G2. The same procedure is repeated 24 hours later but this time the cleaner B is heated in order to recover the residual amount of gas which could not be liberated during evacuation in the cold state.

Sommaire : Un appareil Collins à liquefier l'hélium, du type circuit ouvert, a été transformé en appareil du type circuit fermé, dans le but d'obtenir une reduction appreçiable des pertes d'helium.

Article by
A. van Itterbeek
L. de Greve &
H. Myncke
Appl. Sci. Res.
3, A, No. 6
1953
429-432

III — Vacuum Processing Techniques — III

Abstract No. and References

30 — EVAPORATION — 30

The Evaporation of Metals and Metalloids in Vacuum 1/III

See Abstract No. : 38/III

Metal Finishing by High Vacuum Metallisation 2/III

United States. The application of reflective coatings to die-castings, stampings, slush castings and machined pieces by the vacuum evaporation method has now reached a stage of development where, for reasons of its low production cost and high production output, it is of commercial interest to the metal parts manufacturer normally relying on electroplating for surface decoration of his products. The process is carried out in 3 steps. The first step is undercoating with a lacquer which seals the pores in the surface of the component to be coated and insulates the metal deposit from the component thus preventing corrosive combination. In addition this lacquer must have good adhesion and a low content of volatiles. Drying and setting of the undercoat must be rigidly controlled with regard to moisture content, drying temperature and drying time. The second step comprises the deposition of the metal film. The metal used is aluminium in 95% of all applications because it is comparatively cheap, has a good reflectivity and high resistance to tarnishing. One pound of aluminium facilitates the coating of an area of 25,000 sq. ft. But silver, gold, copper, zinc, chromium, cobalt, nickel, selenium and various alloys are also used. Details of the plant required are given. The pumping unit consists of an oil diffusion pump, an oil ejector pump and a rotary fore pump. The operational pressure should not be higher than 10^{-3} mm. Hg. The third step is the application of a top coat of lacquer. The properties of this lacquer should be identical with those of the lacquer for the undercoat. In addition, it should be highly transparent and suitable for dye-tinting. As it is applied after completion of the vacuum process, its volatile content is less critical. The main purpose of the coat is protection against abrasion. Other applications of the process listed are, the aluminising of reflectors for motor car head lamps and flashlights and the evaporation of selenium in the manufacture of rectifiers and photosensitive elements.

Sommaire : Le revêtement de pièces de métal par la méthode d'évaporation sous vide a maintenant atteind un niveau où il devient d'un intérêt commercial. Des détails sur le procédé sont donnés et certaines applications industrielles sont mentionnés.

Article by G. H. Bancroft *Metal Progress* **64**, Sept., 1953 87-90

Vacuum Process Brightens Plating Picture 3/III

United States. A short account is given of the development of the vacuum evaporation process. Its present scope is briefly reviewed with regard to the finishing of metal components as an alternative to electro-plating, the decorative coating of parts made from non-conducting materials and its uses in the electronics industry. Technical data and production hints on the actual deposition and the ancillary lacquering process are given. The future prospects of the method are discussed. Photographs are shown of a vacuum coating production unit employed for the finishing of metal reflectors. The operating costs of the process are composed of power and labour only as the value of the material deposited is negligible. The type of plant available normally calls for production in batches except in the case of plastic and thin metal sheeting which can now be coated in a continuous operation. The use of the recently introduced rotary gas ballast pump in pumping units of coating plant have eliminated many problems which caused long pumping-down times in the past.

Sommaire : Un compte-rendu de l'importance industrielle jouée par le procédé d'évaporation sous vide dans le domaine varié de la technique et de la décoration.

Article by G. A. Goetz *Steel* 17.8.1953

Vacuum Metallising and Plating—A Comparison 4/III

United States. The author evaluates the scope of the vacuum metallising process and the electroplating process from the practical point of view of a manufacturer. The technical requirements and the production procedure of the vacuum process are analysed in detail. Apart from capital investment, in the case of finishing metal components, the following sequence of operations is given : Clean (phosphate lightly for maximum adhesion and dry work); Rack; Lacquer (First coat, *i.e.* Fill-in coat); Bake; Lacquer (second coat to impart gloss); Final Bake (prior to evacuation); Metallise; Lacquer (Top coat for protection); Bake; Unrack. In the case of plastic components the sequence is given as : Rack; Wash (in a suitable solvent); Lacquer; Bake or Dry; Metallise; Lacquer (for protection); Bake or Dry; Unrack. The plant required by an electroplater involves little expense. A few tanks, gas burners, a generator or rectifier, copper wire for stringing and drums to hold the material for water dip lacquering. As long as the customers' requirements with respect to the quality of the finish are satisfied by this form of lacquering and, may be, an additional burnishing operation, the electroplater who finishes metal components can hold his own. As soon, however, as the client asks for a quality of finish necessitating proper buffing operations, the vacuum metallising process, despite the heavy capital outlay, is more economical provided a sufficient volume of work is in hand. In the case of finishing plastics components the vacuum process is indisputably the most advantageous. In the case of metal components, while the choice of the method is governed by the ancillary hand operations required in each case, it is stated that vacuum metallising cannot compete with bulk finishing as done by the plater and will never replace electro-plating completely.

Sommaire : La métallisation sous vide et le recouvrement par electrolyse sont comparés de point de vue des fabricants et on a trouvé que les coûteuses opérations de polissage font que ce dernier devient d'autant plus onêreux que la quantité à exécuter est grande.

Article by I. Cross *Metal Finish* Dec., 1953 59-63 & 78

Vol. 4 1954

III — Vacuum Processing Techniques — III

Contd.

Abstract No. and References

Economical Coatings by Vacuum Metallising

5/III

United States. Vacuum metallising, particularly of plastics, has become an important mass production method. The plant required is compact and can easily be fitted into an assembly line. The common coating material is aluminium of normal commercial quality. Short ends of aluminium wire 0.07 inch in diameter and about ½ inch long are placed into a tungsten filament which serves as the evaporation source. The aluminium evaporates after heating it to 1,832°F at a pressure not exceeding 10^{-2} mm. Hg. The optimum operational pressure is 5×10^{-4} mm. Hg. The deposition takes 2–3 minutes and the deposit obtained has a thickness ranging from 0.00001–0.001 inch. Pumping-down requires about 30 minutes. Jigs and fixtures suitable for mass production are shown in illustrations. Details of the lacquering technique are given. Means of obtaining colour effects are described. One company is reported to have succeeded in dying metallised components in three colours. In some instances the metal is deposited on the rear surface of transparent plastics. This eliminates the operation of applying the top coat of lacquer. The process is also used for the coating of components made from ordinary metals with precious metals such as gold and silver. The price of a metallising plant depends on the size of the coating chamber and varies from $20,000 to 40,000. Major factors of the three existing finishing methods are compared in a table reproduced below referring to the same production item in each case :—

Comparison of Industrial Metallising Processes

	Silver Reduction	*Electroplating*	*High Vacuum*
Quantity Produced	15,000 units	15,000 units	15,000 units
Appearance	Bright, clean Dulls with age	Bright, slightly rough May dull with age	Bright, clean Remains bright
Wearability*	Good	Excellent	Good
Time Required	9 man days	8 man days	3 man days
Cost per Unit	$.07	$.13	$.03

* Topcoating used on silver reduction and vacuum methods

Various applications of the method are described. In one case a torch manufacturer discontinued the manufacture of polished aluminium reflectors with a reflectivity of 62% and substituted aluminised plastic reflectors which had a reflectivity of 85%.

Sommaire : On discute quelques aspects techniques et commerçiaux de la métallisation sous vide; une comparaison du prix de revient de différents procédés de finissage est donnée.

Article by M. Shepard *Mater. & Meth.* 37, June 1953 97-99

A Study of the Propagation Mode for Metallic Vapours in Shadow Casting by Vacuum Evaporation of Au^{198} and Cr^{51}

6/III

See Abstract No. : 33/I

The Origin of Stress in Metal Layers Condensed from the Vapour in High Vacuum

7/III

United Kingdom. The origin of stresses in electrodeposits has been the subject of many investigations but little is known yet on the amount of the stress and the nature of the stress, *i.e.* tensile or compressive, which occurs in electrodeposits. In order to obtain more information the stress occurring in deposits obtained by vacuum evaporation has been investigated. The following metals were studied : Iron, nickel, palladium, copper, gold, silver, antimony, aluminium, magnesium, bismuth and zinc. The metal to be tested was deposited on a copper strip, 3.4 cm. long, 0.97 cm. wide and 0.0155 cm. thick, one end of which was clamped to a support, fitted to the base plate of a bell jar. The face of the strip was in the vertical plane and the axis in the horizontal plane. The deflection of the free end of the strip could be observed through a travelling microscope. The evaporation was carried out from a filament, as long as the copper strip, placed at a distance of 5 cm. from it. A heater coil was provided at the back of the copper strip. The metal deposits were 1,000 to 3,000 Å thick. Stress occurring in the deposit was indicated by the deflection of the free end of the strip and could be calculated by a formula developed previously by Brenner and Senderoff :

$$S = Et^2 \delta / 3dl^2$$

where S is given in kg/sq. cm., the deflection, δ, in cm., the thickness, t, and the length, l, of the strip in cm and the thickness of the deposit, d, in cm. The value of E, Young's modulus of the strip applicable to the experimental conditions was 6.5×10^5 kg/sq. cm^2. The deflection od the strip during the experiments indicated tensile stress in all but one case and ranged from 0.01 to 0.05 cm. The results of the experiments are shown in a table partly reproduced on next page. The melting points and recrystallisation temperatures of the metals tested are inserted in the table. From an examination of these values it appeared that the stress observed was related to the degree of mobility of the freshly deposited atoms, governed by the temperature prevailing at the face at which they were deposited. The nickel, iron, palladium, copper, gold and silver deposits consisted of crystals of 100 to 150 Å diameter randomly disposed or weakly orientated with a common axis towards the direction of the vapour beam, whereas antimony, magnesium, bismuth and zinc deposits consisted of crystals bigger than 400 Å diameter showing a marked orientation with a net plane parallel to the substrate. These two crystalline structures are known to indicate low and high atomic mobility in the fresh deposit respectively. In view of these results a separate series of experiments was carried out evaporating

III — Vacuum Processing Techniques — III
Contd.

Abstract No. and References

Observed Stresses in the Metal Deposits Condensed from the Vapour 'in Vacuo,' and Some Associated Properties

Metal	*Approx. Mean Observed Stress (kg./sq. cm)*	*Melting Point (°C)*	*Recrystallisation Temp. (°C)*	*α Coefficient of Linear Expansion*	*Young's Modulus E (dyn/sq.cm)*	*Temp. Range Where Stress Occurs △T (°C)*	*Est. Theoretical Stress (kg./sq. cm)*
Iron	3100	1539	350–450	12×10^{-6}	21×10^{11}	200–300	~ 3000 *
Nickel	3500	1455	530–660	14×10^{-6}	21×10^{11}	380–500	~ 3000 *
Copper	900	1083	200–230	17×10^{-6}	12×10^{11}	80–110	~ 1000 *
Gold	850	1063	200	14×10^{-6}	8×10^{11}	80	900
Zinc	0	419	20	26×10^{-6}	9×10^{11}	0	0

* Above the elastic limit

nickel on copper strips, kept at room temperature and 125°C respectively. The stress measured in the latter case was reduced to 75%. It is concluded that the tensile stress observed is originated during the cooling of the deposit. While the metal crystals in the deposit grow as a result of freshly arriving atoms during evaporation, thermal expansion of the crystals takes place. This expansion is checked and contraction initiated as the thickness of the deposit continues to grow and the temperature of the deposit at the level considered is progressively decreasing until it falls eventually below the recrystallisation temperature. At this stage mobility of the atoms is nil and where readjustments are not completed tensile stress will develop throughout the deposit on further cooling. Its actual value depends therefore on the difference between the final deposit temperature and the recrystallisation temperature. Aluminium was the only metal which showed compressive stress. This is attributed to oxidation occurring during the growth of the deposit.

Sommaire : La nature et la valeur de la tension dans des dépôts évaporés, d'une variété de métaux, ont été étudiées. La tension est indiquée, sa valeur étant gouvernée par les conditions de température à l'intérieur du dépôt, pendant le procédé de déposition.

Article by H. P. Murbach & H. Wilman *Proc. Phys. Soc.* **66B**, Nov. 1953 905-910

The Activities of Evaporated Metal Films in Gas Chemisorption — 8/III

See Abstract No. : 44/I

Thin Films of Ferromagnetic Materials — 9/III

United States. Experimental observations are reported on the magnetic properties of thin evaporated films of nickel. The saturation magnetisation follows the general prediction of Klein and Smith after somewhat arbitrary modifications on the basis of fitting the Weiss curve for bulk specimens, as suggested by Drigo. Qualitatively observed properties due to domain structure follow the preductions of Kittle. The surface roughness of the specimens has been carefully controlled. Measurements of temperature coefficient of resistance have led to modification of the theory of electron mean free path in thin films. The mean free path is found to vary at small thicknesses, rather than remaining constant as assumed by Fuchs and Sondheimer. The surface roughness has been estimated with the help of this theory. The films of nickel behave as if surface variations extend into the film approximately 3Å from each surface.

(*Science Abstracts*)

Sommaire : Des observations expérimentales sur les propriétés magnétiques de couches fines de nickel, évaporées sont rapportées.

Article by E. C. Crittenden, Jr., R. W. Hoffman *Rev. Mod. Phys.* **25**, Jan. 1953 310-315

Sorption Properties of Thin Nickel Films — 10/III

See Abstract No. : 45/I

Structure of Evaporated Films as a Function of Film Thickness — 11/III

United States. It is known that the structure of evaporated bismuth films on glass depends on the film thickness. Their structure below 160 Å thickness had been found to be discontinuous, the crystals of the deposit growing as the layer thickness grows and the electrical conductance of the layer being constant until a critical thickness value is reached, where the latter changes rapidly. Since these results became known the radio-active tracer method of film thickness determination has been developed which facilitates extreme accuracy of measurement (± 4%) and the present work was undertaken to conclude and confirm previous findings using this technique on films ranging in thickness from 2–300 Å. Three different methods of investigation were employed. The external structure of the film was examined in the electron microscope, the internal structure

III — Vacuum Processing Techniques — III
Contd.

Abstract No. and References

in the electron diffraction camera and the electrical film properties were studied by measuring their conductance. Details and results of all experimental procedures are given. The particle size of the films, as determined in the electron microscope, is shown in a table partly reproduced below :—

Bismuth Film Particle Size Determination and Related Data

Film Thickness (Tracer Method)	*Average Particle Diameter in Å*	*Particles Per Sq. Micron Population*	*Per Cent. Surface Covered*	*Film Thickness (Particle Size Method)*	*Ratio of Thickness Column 5/ Column 1*
1	2	3	4	5	6
23.8	236	520	22.5	51.9	2
74.6	450	261	41.4	186.3	2
91.7	628	234	71.8	450	5
115	667	181	62.6	420	3
203	815	140	72.7	594	2

The specimens of the films to be examined in the electron microscope were coated with 1% collodion in amyl acetate, dried in an evacuated desiccator for 2–4 days in order to ensure stability during examination and then stripped off the glass substrate with Scotch tape but this stripping process was successful only with films up to 100 Å thick. Films below about 10 Å thickness showed a ' fern-like ' structure of the crystal aggregates in the electron micrograph. This is suspected to be a replication of the underlying glass dendritic crystals. The electron diffraction studies were carried out by the reflection and the transmission method. The information obtained by both methods was conplementary and revealed pronounced orientation effects with the dominant orientation of the (200) planes parallel to the glass substrate in the case of films 100-500 Å thick. No accurate information could be obtained in films less than 100 Å thick. A special specimen holder was used in these experiments which could be rotated about an axis perpendicular to the direction of the beam and facilitated the tracing of orientation effects. The results of the electron microscope and diffraction camera observations coincide with the conductance measurements, indicating that the discontinuous film structure changes to a continuous structure at thicknesses approaching 100 Å. The conductance of films from 500 Å upwards is approximately that of the bulk metal. In the thickness range from 2–75 Å the conductance is virtually constant. The range of rapid changes extends from 90–125 Å thickness, the conductance measuring 2.1×10^{-14} mho at 90 Å and 1.4×10^{-3} mho at 125 Å. The bismuth samples subjected to conductance measurements were prepared by first evaporating platinum strips on the glass plate to serve as electrodes. Subsequently the gap between the two platinum strips, 0.16 cm. wide, was filled in by depositing the bismuth.

Sommaire : Une étude complète sur les propriétés de couches minces de bismuth faite en employant la méthode du traceur radio-actif pour la détermination de l'épaisseur des différents films étudiés.

Final Report by P. J. Bryant H. U. Rhoads, & A. H. Weber *Nat. Sci. Foundation Grant-150 University St. Louis* September 1953

Fine Structure in Polyethylene Terephthalate Fibres 12/III

See Abstract No. : 23/II

Refinements in the Preparation and Theory of Multilayer Films 13/III

See Abstract No. : 28/I

Method and Equipment for the Deposition of a Metal Layer on Wire or Similarly Shaped Material 14/III

Liechtenstein. A plant is described which has been developed for the continuous coating of wires or similar material and is particularly suitable for the coating of fine wires with precious metals such as are required, for instance, in the manufacture of grids for thermionic-valves. The plant consists essentially of a large chamber which is evacuated. In the centre of the chamber is a small special processing enclosure. The latter has the form of a cylinder, closed at both ends and positioned vertically. The cylinder is split horizontally in the centre and the two components are fitted with a flange. The two flanges have cavities just large enough to let the wire to be treated pass. In operation the wire is wound off one drum, passed through the processing enclosure and wound on to another drum. Both drums are situated in the large chamber. The metal to be deposited is placed into the processing enclosure prior to its assembly and is evaporated by heating the processing enclosure as a whole. If resistance heating is used the enclosure is conveniently constructed from graphite. If high-frequency heating is used the processing enclosure could be quartz or a suitable refractory. The inner surfaces of the processing enclosure should have a temperature higher than that of the evporated metal in order to prevent condensation on these faces as this may result in the formation of droplets which may fall on to the wire. At the same time, this measure facilitates great economy in the use of the material employed for coating. The wire, on its way to and from the processing enclosure, runs through a solenoid situated in the chamber. This is arranged to clean the wire prior to coating and to effect diffusion of the deposit into the body of the wire after processing in order to ensure good adhesion.

Sommaire : Description est faite d'un appareil qui facilite le revêtement uniforme de fils fins par la méthode d'évaporation ,d'une manière qui guarante l'utilisation très économique du matériau evaporé.

A. Vogt *Swiss Pat.* 288,438

III — Vacuum Processing Techniques — III
Contd.

Abstract No. and References

Fabrication of Radio-Frequency Micropotentiometer Resistance Elements 15/III
See Abstract No. : 22/I

The Production of Diffraction Gratings 16/III
See Abstract No. : 27/I

The Behaviour of Light-Sensitive Evaporated Layers in the Electron Microscope 17/III
See Abstract No. : 19/II

Photoconductivity of Indium-Selenide 18/III
See Abstract No. : 31/I

Photographic Copying Process 19/III
See Abstract No. : 62/I

31 — CATHODIC SPUTTERING — 31

The Photoelectric Effect of Thin Bismuth Films 20/III

United States. Thin Bismuth films on glass were obtained by sputtering in argon. The photoelectric currents were measured when ultraviolet light was focussed on a film, and at the same time direct current passed through the film in one direction and also in the reversed direction, as well as alternating current. It was found that only relatively large alternating currents affected the photocurrent—thus heating alone did not affect the photocurrent much. The direction and magnitude of the direct currents, however, affected the photocurrent appreciably.

(*Science Abstracts*)

Note by D. J. Fourie *Phys. Rev.* **91**, 15.8.1953 803

Sommaire : L'effet photo-électrique de couches fines de bismuth, obtenues par projection cathodique dans l'argon a été l'object de recherches.

Sputtering of Stainless Steel by Protons in the 30–80 keV Range 21/III

United States. The sputtering of metals caused by hydrogen ion bombardment has been investigated before. These experiments seemed to indicate that the number of sputtered atoms per incident ion increases linearly with the energy in the range up to 500 eV. The authors conducted a similar investigation, reported here, in the range 30–80 keV. They employed a beam of $70\%H_1^+$ and $30\%H_2^+$. The target, $\frac{1}{8}$ inch thick and 4 inch. in diameter, was of stainless steel. The mass of material sputtered was determined by weighing the target before and after each run. The pressure in the chamber was 10^{-5} mm. Hg. The accelerating voltage was monitored with the help of an oscilloscope. The measured values of the accelerating voltage and current to the target for a given period gave the number of incident particles and their energy. As a check the current was calculated from the amount of heat removed by the water cooling the target during the bombardment. The formula used for this calculation is given. The total ampere–hours of beam, hitting the target in a run, were determined by establishing the values of the current at regular time intervals, plotting these values against time, cutting out the resulting area of the graph paper and weighing the paper on an analytical balance. The results of the experiments are shown in a table reproduced below.

Results of Hydrogen Ions on Stainless Steel

Accel. Voltage (kV)	*Mass Before Bombardment (g)*	*Mass After Bombardment (g)*	*Mass After Nitric and Chromic Acid Wash (g)*	*Mass Diff. (g)*	*Amp. Sec. Total ±15%*
36.3	409.410	409.404	409.396	0.016	3020
50.9	403.528	403.511	403.508	0.020	2220
74.9	409.289	409.268	409.286	0.021	3460

The ratios of sputtered atoms to incident ion were calculated for each energy value as follows : 0.92×10^{-2} at 36.2 keV; 1.6×10^{-2} at 50.9 keV and 0.97×10^{-2} at 74.9 keV. Washing the target after a run in 16 N nitric and chromic acid resulted in a reduction of its weight. This seems to indicate the formation of a deposit during

Vol 4 195

III — Vacuum Processing Techniques — III

Contd.

Abstract No and References

bombardment. The experiments which have been of a preliminary character proved that ions of high energies do not cause as much sputtering as ions of low energy. The high-energy ions seem to bury themselves temporarily in the target. It is recommended to carry out experiments in the 5–10 keV range which is likely to prove to be the range of peak sputtering.

Sommaire : Une étude préliminaire des taux de projections de protons bombardant une cible d'acier inoxydable dans la gamme de 30 à 80 keV, a été faite expérimentalement.

Report by F. Fairbrother Jr. & J. S. Foster Jr. *Univ. Calif. Rad. Lab. Report No.* URCL-4169 Aug. 1953

36 — DRYING — 36

Moisture Removal 22/III

See Abstract No. : 8/I

Design and Operating Technique of a Vacuum Drying Oven. Part I. Design of the Oven 23/III

United Kingdom. The design of a vacuum oven is described which can be used for the same purposes as a refractometer and/or for the drying of such labile products as sugar syrups and molasses. The design incorporates certain features developed in answer to criticisms of oven constructions voiced in the Proceedings of the International Commission for Uniform Methods of Sugar Analysis, 1949. The oven consists essentially of a thick circular baseplate with a shallow rim holding 6 sample dishes in a position very close to each other. The oven is closed by a heavy lid clamped down over a central spigot. The lid carries a silicone rubber ring fitted into a circular groove. This makes contact with the raised annulus of the base when the oven is closed and forms an airtight seal. Air admitted to the oven enters through 6 ports in the base, inclined at an angle of 45°, so that the air reaches the interior of the oven just above the dishes holding the material processed. It is removed through ports provided in the lid inclined at 30° and positioned in the central area of the lid surrounding the spigot. The air flow can be adjusted from 0.5 to 100 ml./min. by means of a needle valve. The air is dried by passing it through activated alumina and over barium oxide lumps contained in a tubular drying train which forms part of the oven unit. After passing the drying train the air contains a maximum of 0.0007 mg./l. moisture. The heat is supplied from 12 electrical heaters with a total wattage of 80 arranged radially around the base. At normal reduced pressure the operational temperatures should range from 50 to 90°C and at atmospheric pressure temperatures up to 105°C. can be employed. It is advisable to raise the temperature slowly in order to prevent crust formation. The temperature is automatically controlled by a thermostat. A series of drying tests on cane molasses agreed to within ±0.05 % loss.

Sommaire : Description d'un four symétrique de forme circulaire d'une grande capacité pour séchage sous vide, avec distribution uniforme de la température.

Article by S. D. Gardiner *Analyst* 78, Dec. 1953 709-711

Moisture in Refrigerant Machinery Circuits and Components 24/III

United Kingdom. 90% of the troubles encountered in the operation of refrigeration plant using Freon–12 or methyl chloride as the refrigerant are believed to be due to the presence of excessive moisture. Excessive moisture can cause freeze-ups at the expansion valve or in the low side of refrigeration plant and may produce a complete blockage so stopping the plant. The solubility of moisture in the halogenated refrigerants is extremely low and an acceptable limit for Freon–12 is 10 p.p.m. Still lower limits are often set for sealed units. Ammonia and carbon dioxide equipment is less liable to give trouble on this account. Ammonia can accommodate large quantities of moisture by absorption and the solubility of water in carbon dioxide is high. In the case of sulphur dioxide equipment the solubility of water in the refrigerant is also very high but the limit is set by corrosion. Corrosion due to the presence of moisture develops in all types of refrigeration equipment but in sulphur dioxide plant it develops particularly rapidly. Moisture in refrigerating plant can do harm in other directions. For instance, it has been found that compressors in refrigerating plants charged with Freon-12, methyl chloride or other halogenated refrigerants seize-up because the moving parts become 'copper-plated'. This is a complex chemical reaction which may be started by moisture but, at any rate, is seriously aggravated by it. Moisture may enter the refrigeration circuit by eight different means. The symptoms of its presence in the circuit are listed and advice is given on the precautions to be taken in order to prevent entry of moisture during the manufacture, erection and operation of plant. A simple test for detecting as little as 10 p.p.m. of moisture in refrigerator oil is described. The precautional measures are much simpler than the remedial measures required afterwards. There are various dehydration methods available for the removal of moisture. Where the amount of moisture to be removed is small, desiccants of the type most effective at low temperatures are employed but these must be replaced or reactivated at frequent intervals. Two commonly used desiccants are activated alumina and silica gel. Detailed information is given on the respective properties of these materials and comparisons are made with the performance of other dehydrating agents in a table which is reproduced on next page. Where large quantities of moisture have to be removed vacuum or inert gas dehydration methods are employed. There is a variety of procedures carried out in vacuum or in an inert gas atmosphere such as drying by heating, by compression and by expansion or direct drying by pumping the air through a refrigerated auxiliary vessel or a vessel containing a desiccant material. Essential details on all these procedures and on the

III — Vacuum Processing Techniques — III
Contd.

Abstract No. and References

Material	*Residual water mg. per litre*	*Equivalent dewpoint °F.*
Calcium sulphate $CaSO_4$ (anhydrous)	2.8	+ 19.4
Calcium chloride $CaCl_2$ (granular)	1.5	+ 10.4
Calcium chloride $CaCl_2$ (tech. anhydrous) ...	1.25	+ 1.4
Zinc chloride $ZnCl_2$ (sticks)	0.98	— 2.2
Barium perchlorate $Ba(ClO_4)_2$ (anhydrous) ...	0.82	— 4.0
Sodium hydroxide NaOH (sticks) caustic soda ...	0.80	— 5.8
Calcium chloride $CaCl_2$ (anhydrous)	0.36	— 20.2
Magnesium perchlorate $Mg(ClO_4)_2$ $3H_2O$ ...	0.031	— 58.0
Potassium hydroxide KOH (sticks)	0.014	— 68.8
Silicon dioxide SiO_2 silica gel	0.006	— 79.6
Calcium sulphate $CaSO_4$ (anhydrous) Drierite ...	0.005	— 83.2
Calcium oxide CaO	0.003	— 88.6
Magnesium perchlorate $Mg(ClO_4)_2$ (anhydrous)	0.002	— 94.0
Aluminium oxide Al_2O_3 activated alumina ...	0.001	—102.0
Barium oxide BaO	0.00065	—106.6
Phosphorus pentoxide P_2O_5	0.00002	—142.0

Information mainly derived from U.S. Bureau of Standards Research Paper RP 1603

respective plant required are given. Due reference is made to mass production conditions and repair-shop practice. The paper concludes with a list of points which require special attention when vacuum dehydration processes are employed.

Sommaire : Les problèmes relatifs à la pression, les effets et la suppression de l'humidité contenue dans les circuits de travail d'un appareil de réfrigération sont discutés. Des détails sur les procédés de déshydration sous vide, appropriés à la suppression d'humidité, sont donnés.

Article by M. B. Ranken *Mod. Refrig.* *beginning* June, 1953, 203 *ending* Nov. 1953, 408

Vol 4 195

Puffs Foods Continuously in Special Vacuum Process

25/III

United States. Equipment is described which has been built to make lightweight puffed confectionery. Some products are expanded to 30 times their original volume. The merits of the process to the production of certain cereals are being investigated as it permits the cooking of the cereals at a comparatively low temperature which assists the preservation of the food value. The puffing plant consists of a tunnel, 100 ft. long and 18 inch. in diameter which is evacuated to 5 mm. Hg by means of 2 rotary pumps and is fitted at each end with a pair of rotary vacuum lock valves, served by a separate vacuum pump to facilitate loading and discharging respectively. An endless conveyor belt carries the product through the tunnel. The belt and the vacuum lock valves are driven by variable speed motors which are controlled separately in order to adjust the flow of work to suit the individual product. The product to be puffed should have a minimum moisture content, should be hot enough to ensure good plasticity (140–220°F) and should be formed into small pillows or balls. The processing (puffing) time varies from 1 minute to 3 minutes according to the material. The finished puffed product should not be handled or stored in a humid atmosphere. The plant described has a production capacity of 4,000 lb. of ' Mintlets ' per 8-hour day.

Sommaire : Description d'un appareil à vide pour le soufflage, en confiserie, sur une grande échelle commerçiale.

Article by I. C. Miller *Food. Engng.* **25**, Dec. 1953 68-69

Puffed Corn Production and Method of Making the Same

26/III

United States. A vacuum method is described for the manufacture of puffed corn suitable for French frying which eliminates the risk of caramelisation and yields a tasty end-product of crisp, chewy texture. Cracked waxy maize or field corn, each granule reduced to about half the original kernel size, is placed in a sealed vessel which is pumped down, and kept at approximately 0.5 inch Hg for about 2 minutes in order to degas the corn. Then steam is introduced into the chamber at a pressure of 100 lb./sq. inch. to condition the material for the puffing process. After 2 minutes the steam pressure is reduced rapidly to 50 lb./sq. inch. This is followed by an instantaneous reduction in pressure to about 0.5 inch Hg. This causes an explosion resulting in puffing of the cracked corn to about $3\frac{1}{2}$ times its original size. The reduced pressure is maintained until the corn is cold set. During the steaming operation condensed water and non-condensable gases are removed *via* a purge line. The material is actually puffed in a puffing chamber which is much larger than the steaming chamber. The communication between the two is of a diameter substantially the same as the size of the steaming chamber.

Sommaire : Une méthode employant le vide pour la production de céréales soufflés appropriée pour la torréfaction à la mode Française de graine de maïs.

C. R. Carman & Guardite Corp. *U.S. Pat.* 2,653,104

The Recovery of Essence from Florida Orange Juices

27/III

See Abstract No. : 46/III

New Volatile Flavour Process

28/III

See Abstract No. : 47/III

III — Vacuum Processing Techniques — III
Contd.

Abstract No. and References

37 — METALLURGICAL PROCESSES — 37

Metal Melting under Vacuum. Some Mechanical Problems Involved 29/III

See Abstract No. : 54/I

Developments of Commercial Vacuum Furnaces for Melting Metals and Alloys 30/III

See Abstract No. : 14/I

Improvements i.o.r.t. Cleaning Observation Windows in High Vacuum Chambers 31/III

Liechtenstein. During the melting of metals in a vacuum furnace vapours are evolved which condense on the observation windows provided to facilitate visual inspection of the process. These layers soon become opaque. In the past, in order to overcome this difficulty the observation window was protected on the chamber side by a second glass plate which could be exchanged with a clean plate by means of a mechanical device without interrupting operations, when the layer had reached opacity. But the number of exchanges possible was restricted and, therefore, the usefulness of such a device was limited. A new device is proposed employing only one protective glass plate in a permanent position. Its inner surface is cleaned periodically by an externally actuated brush consisting of metallic bristles and fitted to one edge of a sector-shaped plate of a size larger than the diameter of the observation window. The plate is mounted on a shaft running in a vacuum seal through the wall of the chamber and carrying a handle at the external end. When, during processing the observation window is not required the plate is moved over the window masking it completely and thus preventing deposition of condensate on it.

A. Vogt *Brit. Pat.* 699,921

Sommaire : Description d'un appareil pour nettoyer les fenêtres d'observation de fours à vide qui permet d'enlever les vapeurs deposées par brossage.

Furnace for Vacuum Distillation of Zinc and Silver, Constructed by the Institute Pesquisas Tecnologicas of Sao Paulo 32/III

Spain. The author describes in detail the furnace and its operation for vacuum distillation of zinc contained in alloys of zinc and silver (1) which result from the liquefaction of Parkes' crusts. 1,720 kg. containing 22% silver and 65% zinc is charged per day to 6–9 horizontal retorts made of cast iron, containing 2–3% chromium. Some retorts after 550–740 hours of operation are still in good shape.

(*Chemical Abstracts*)

Article by Th. D. de Souza Santos *A BM* 9, No. 30, 1953 141-159

Sommaire : Description d'un four de distillation du zinc et de l'argent, construit à l'Institut de Recherches Technologiques de Sao Paulo.

Vacuum Dezincing of Desilvered Lead Bullion 33/III

United States. The theoretical aspects of vacuum dezincing according to the process described in Vol. III, Abstract No. 37/III are discussed. Operating under reduced pressure the volatile constituents of molten metals evaporate from the exposed surface only, and the rate of evaporation depends on the surface area, the surface temperature, the surface concentration of the volatile constituents and their partial pressures immediately above the surface. The distillation rate does not depend only on the evaporation rate but also on the ability of the molecules evaporated to reach the condensing surface. Thus the distillation rate should be highest under the conditions of molecular distillation. In all other cases, back condensation will take place and the net evaporation rate will be smaller than its intrinsic value, i.e. equal to the condensation rate. These fundamental relationships are considered with respect to the process in question which is operated both as a batch and as a continuous process. In the latter case the path of the evaporated molecules is horizontal, the distillation space about 30 cm and the operational pressure about 100 micron Hg. The bullion from which the zinc is recovered flows through the apparatus in the form of a falling film. A formula is developed, based on Langmuir's equation for absolute rates of evaporation, which relates E, the net distillation rate with p_1, the partial pressure of the volatile element in the distilland, V, the operational pressure, and a, b and c, constants depending on the state of the distilling element, the distillation path and the condenser surface respectively:

$$p_1 = V + c + \frac{2E}{a} - V \exp(-E/b)$$

For practical purposes this formula can be approximated, expressing the distillation rate by $E = a(w - rV)/2r$, where $a = 0.472/\sqrt{T}$, T being the temperature in °K of the evaporating surface, w is the weight per cent of zinc contained in the bullion, r is the ratio of w/p_1. Further elaboration of these formulae permits the calculation of treatment times required at various pressures and for the two cases of cold and liquid zinc condensation. The results are shown in graphs and agree reasonably well in the case of the continuous process. But in the case of the batch process the calculated value is 1,460 seconds and the practical time is 18,000 seconds. This has been found to be due essentially to pre-condensation phenomena. A certain amount of lead is evaporated at the same time as the zinc and after saturating the distillation space, tends to condense into liquid globules,

III — Vacuum Processing Techniques — III
Contd.

Abstract No. and References

on to which some of the zinc vapour condenses during flight. In the case of the continuous process, these liquid globules eventually reach the lower surface of the condenser but in the case of the batch process the path of the vapour is vertical and the liquid globules are returned to the evaporating surface. Methods of calculation and results are also given for the determination of absolute evaporation rates, mean velocity of the zinc vapour, the percentage of lead in the distillate and the latent heat of vaporisation of zinc.

Sommaire : Les aspects théoriques d'un procédé de dezincage sous vide de lingot de plomb (voir Vol. III., Resume No. 37/III) ont été étudiés.

Paper by T. R. A. Davey *J. Metals* Aug. 1953 991-997

The Production of Pure and Deliberately Contaminated Iron. Experimental Investigations into the Properties of these Metals

34/III

Germany. The study of certain properties of steels, such as ageing phenomena is difficult because steels normally contain other substances in appreciable amounts, for instance manganese, carbon, sulphur, phosphorus, oxygen, nitrogen and hydrogen which will affect the results of such investigations. Therefore, in order to facilitate respective experiments, the author had first of all to prepare samples of iron as pure as possible and alternatively samples of iron which were deliberately contaminated. The raw material was the purest commercially available carbonyl iron powder, grade L, marketed by the General Aniline and Film Corporation in New York. The only contaminants traceable in that material were carbon, oxygen and nitrogen. These substances had to be removed by hydrogen and high vacuum treatment. The iron was vacuum melted in a specially designed furnace which permitted the casting of an ingot of about 2 kg. The construction material was glass and heating was effected by a 25kW induction heater, operating at 300,000 c.p.s. The pumping plant consisted of an oil diffusion pump of 500 l./sec. backed by a fore pump producing an ultimate pressure of 10^{-5} mm. Hg. The melting crucible was made of aluminium or magnesium oxide and was situated in a quartz container which in its turn was supported by a quartz tube. The space in-between was filled with magnesia. Casting was carried out through a hole in the bottom of the crucible and the surrounding container which could be closed with a rod made from magnesia and strengthened by molybdenum. The rod was moved by magnetic action. The mould was attached to the baseplate and had a jacket to facilitate water cooling. The iron samples were passed from a storage space into the crucible through a funnel by magnetic means. The whole assembly was covered by a bell-shaped hood of hard glass and was surrounded by an open glass cylinder, filled with cooling water. The samples used had the shape of rods which were vacuum sintered and had absorbed a known quantity of oxygen in the process. After melting down the iron, hydrogen was introduced into the furnace in order to remove all oxygen from the iron. A specially developed indicator was employed to record the progressive elimination of oxygen. With the indicator at 0, the total content of carbon, oxygen and nitrogen had fallen below 0.0001% but at this stage dissolved hydrogen was still present in the melt and this was removed by continuous pumping at 10^{-3} mm. Hg taking the necessary precautions to avoid eruptive evolution of the gas. In operations concerned with adding other elements to the melt, materials such as nickel and molybdenum, having about the same affinity to oxygen as iron, were placed in the crucible prior to the start of the melting process but where the affinity of the materials was higher, for instance, in the case of manganese, chromium, aluminium, the additions were made after purification was completed. Similar information is given on the procedure of adding highly volatile elements or gases to the melt. In spite of great care contaminants are often introduced by the material of the crucible. Generally speaking the purification of the iron is most satisfactory if a magnesium oxide crucible is used. An indication of this is given in a table reproduced below which at the same time shows the results obtained by the methods described.

The Contamination of Iron Melted in an Alumina and a Magnesia Crucible

Element	*Composition of the Iron Melted in Alumina Crucible*	*Magnesia Crucible*
Carbon %	0.000	0.000
Silicon %	0.002	0.002
Sulphur %	0.000	0.000
Aluminium %	0.027	0.001
Cobalt %	0.001	0.000
Copper %	0.002	0.000
Magnesium %	0.000	0.001
Nickel %	0.006	0.003
Oxygen %	0.000	0.000

The article gives also information on the behaviour of iron and steel purified as described with particular reference to ageing phenomena, case-hardening and brittleness.

Sommaire : Examen des propriétés du fer et de l'acier extrèmement purifiés, et détails de la méthode employé pour la préparation des échantillons à examiner.

Article by J. D. Fast *Stahl u. Eisen*, **73**, 5.11.1953 1484-1497

Production of Titanium Metal

35/III

United States. Most of the conventional methods of producing titanium metal by way of reduction of titanium tetrachloride with a metal such as magnesium yield as the end product titanium metal sponge solidly fused with the chlorides of the reacting metal. In order to recover the material from the furnace it has to be broken up into small lumps and the lumps have to be subjected to leaching treatment in order to free the

III — Vacuum Processing Techniques — III
Contd.

Abstract No. and References

titanium metal from the chlorides of the reacting metal. In the process described here the pure titanium metal is obtained directly. The apparatus employed has the shape of a retort with a condensing zone at the top, a furnace-heated reaction chamber in the centre and a receiving zone at the bottom. The receiving zone and the reaction chamber are separated by a tray-like structure restricting the aperture between both zones to an annulus ranging from 0.01 to 0.1 inch. width. In operation, magnesium billets are introduced into the reaction chamber through one opening in the condensing zone and titanium tetrachloride vapours through another. The titanium metal liberated in the reaction is retained on the tray and the magnesium chloride is drained through the circular gap into the receiving zone, where it solidifies. Subsequently, the small amount of magnesium chloride still present on the tray and the unreacted magnesium metal are distilled and deposited on the walls of the condensing zone so leaving the titanium on the tray in the reaction zone. This distillation is carried out at reduced pressure, produced by a vacuum pump connected to the condensing zone. A practical production example is given : 5 lb. of magnesium metal were placed in the reaction zone and the zone was heated to 725°C. Titanium tetrachloride vapours were introduced at ½ lb./min. and 1 lb. of magnesium metal was added every 7½ minutes for 2 hours. About 20% more magnesium than theoretically required was introduced during the operation to ensure complete reduction of the titanium tetrachloride. Subsequently, the distillation was carried out for 16 hours at 640-950°C and the pressure was kept at 10μ Hg for the last 8 hours of the operation. Then the plant was cooled down to 25°C and dismantled. 15.2 lb. of pure titanium were obtained in the operation, *i.e.* a recovery of 95%. Its purity was 99%, its Brinell hardness =130 and its ductility was satisfactory. The receiving zone contained 55 lb. of magnesium chloride. Thus 87.7% of the magnesium chloride formed was drained through the aperture. The condensing zone contained 7 lb. of magnesium chloride and 4 lb. of magnesium metal. An alternative design of the plant, for operation by the same method, has been developed to minimise the difficulties involved in the dismantling of the plant described above. Full details of the alternative design are given.

Sommaire : Une nouvelle méthode de réduction avec du magnésium pour la production de tétrachlorure de titane a été developpée; elle fournit le métal (Titane) directement comme produit fini, éliminant le laborieux procédé auxiliaire de la méthode conventionnelle, donnant le métal pur après la réaction.

C. K. Stoddard
J. L. Wyatt &
National Lead Co.
U.S. Pat.
2,663,634

Improvements i.o.r.t. the Manufacture of Calcium

36/III

United Kingdom. A distillation process to be carried out at reduced pressures is described for the purification of crude calcium. The still, preferably made from stainless steel, is of the pot-type, carrying at the bottom the ingot of calcium to be evaporated. The cover consists of a circular condenser plate slightly smaller in diameter than the width of the still body, leaving an annular gap through which vapours may rise to a second annular-shaped condenser surface situated at a considerable distance above the lower condenser plate. The lower condenser is cooled by compressed air and the upper condenser is water-cooled. A vacuum pump, Pirani-type gauge and means for the admission of argon or other inert gases are provided. In operation, the still with the crude calcium charge is sealed, evacuated and argon at about 2 mm. Hg pressure is admitted. The still is then lowered into a fused salt bath kept at approximately 900°C so that the level of the bath reaches the level of the lower condenser. The latter is cooled to 500°C and the upper condenser is held at 30°–60°C. In these conditions relatively pure calcium is deposited on the lower condenser and the impurities such as sodium condense on the upper condenser. A most effective separation is obtained. As the layer of pure calcium on the lower condenser grows, it may be necessary to cool the condenser to still lower temperatures, say about 300°C after one hour's operation. The pressure should be checked periodically and adjusted when required due to degassing phenomena. When the distillation is complete the still is removed from the bath, and opened when cold. The deposits are removed by a hammer and chisel method. The calcium deposit obtained in the manner described consists of small crystals loosely joined together but if the operation is carried out in a vacuum of say 0.5 mm. Hg the crystals have the shape of long tough needles and it is difficult to break up the layer. If the condenser is of iron or mild steel the face of the calcium deposit in contact with the condenser contains an appreciable amount of iron. This effect is absent, if stainless steel is used. An example is given of the effectiveness of the method as follows :

	Calcium (%)	*Sodium* (%)	*Chlorine* (%)	*Iron* (*p.p.m.*)	*Manganese* (*p.p.m.*)
Crude Calcium	95	0.8	0.65	3010	310
Product of Distillation	99.4	0.12	0.24	52	26

Sommaire : Une méthode de distillation du calcium brut, à basse pression qui assure une séparation nette des impurités à un degré exceptionel.

Article by
R. Le Grice
Burnett &
Ministry of Supply
Brit. Pat.
698,861

Vacuum Fusion Analysis

37/III

United States. The vacuum fusion method of determining gases in metals originally applied to iron and steel, has recently been extended to non-ferrous metals. The results obtained with the various apparatus described in the literature differ. This seems to be due to specific procedures developed in each case to suit the particular apparatus. The work reported here is concerned with the apparatus and method of determining oxygen in chromium using a molten bath of iron to which tin is admitted (25% by weight). A careful description is given of all details and all factors influencing the results of the method. Diagrams of the experimental arrangement and some of its special features are given. The furnace used represents a modified version of the type described by Beach and Guldner. A mercury lift assisted by magnetic action was used to insert the samples

III — Vacuum Processing Techniques — III

Contd.

Abstract No. and References

in the furnace. Non-magnetic samples were wrapped with iron wire. A mercury diffusion pump of the type used by Naughton and Uhlig was employed for the circulation and extraction of gases. It had a speed of 12 l./sec. The evacuation of the whole system was effected by a 3-stage oil diffusion pump made of glass. Instead of stopcocks glass-mercury solenoid valves were fitted. Details of their design are given. The blank rate achieved in the apparatus was 0.1–0.2 litre-micron per minute. Special experiments were carried out, adding a small amount of stopcock grease (Apiezon) to the system in the region near the furnace. The results are shown in a table, partly reproduced below.

Effect of Stopcock Grease on Blank Rate

Set	*Run Description*[a]	*Blank Rate, μ/Min.*
III	N–10	0.201
	G–20	0.236
	G–10	0.227
	N–20	0.190

[a]N, no grease present; G, grease present; 10, 10-minute blank determinations: 20, 20-minute blank determinations.

The procedure employed in the analysis was the following : A synthetic mixture of chromium—chromium oxide was tested in an iron bath or other type of bath at various temperatures; the chromium metal was analysed for oxygen by an independant method. The performance of the fusion method could then be expressed by : $R = \frac{\text{oxygen found}}{\text{oxygen expected}} \times 100$. The values of recovery R (%) of oxygen was then fitted by the method of least squares to the equation $R = a + c_1C + c_2C^2 + t_1T + t_2T^2 + kCT$, where a, c_1, c_2, t_1, t_2 and k are constants, T is the operating temperature and C is the weight % of chromium in the bath. One series of experiments was carried out with a pure iron bath and another with an iron-tin bath, containing 25% (by weight) tin. Better recoveries were obtained with the iron-tin bath operating at about 1,600°C. Recovery deteriorates from 85 to 70% as the concentration of chromium in the bath reaches 5%.

Sommaire : Appareil et procédure de la détermination d'oxygène dans le chrome par la méthode de fusion sous vide employant un mélange de fer et d'étain, comme bain, ont été développés et étudiés avec soin.

Article by W. S. Horton & J. Brady *Analyt. Chem.* **25**, Dec. 1953 1891-1898

38/III

The Evaporation of Metals and Metalloids in Vacuum

Germany. The process of evaporating metals and metalloids in vacuum is extremely useful for the removal of contaminants, for the production of alloys and for the preparation of thin films. The basic laws of the technique are discussed. The governing factors are the melting point, boiling point and vapour pressure of the processed material. The melting point is practically independent of the ambient pressure. The change of this with pressure can be determined by the formula :

$$\sigma = 0.0242 \frac{(V_2 - V_1)\,T}{S} \quad (°K/at.)$$

where V_1 is the volume of 1 gram of material in the solid state, V_2 the volume of 1 gram of material in the liquid state, T the absolute temperature in °K and S is the heat of fusion in cal./g. The order of magnitude of the shift is about 10^{-2}°C for a change of pressure up to 1 atmosphere. Of greater interest is the change of volume as this has a bearing on the size of the crucible to be employed. In the case of aluminium, for instance, the density of the solid material is 2.7 and the volume per gram of the solid material is 0.371. In the molten state, at a temperature of 658°C, the density of the same material is 2.46 and the volume per gram 0.407, whereas the shift of the melting point is only 0.009°C per 1 atmosphere. Normally the vapour pressure of a material at the melting point is negligible but at the boiling point, which as a rule is some 1,000°C above the melting point, evaporation is appreciable. This applies to atmospheric conditions but it is impracticable to evaporate at this pressure because nearly all materials tend to combine with the oxygen in the air. Operation in a protective atmosphere such as hydrogen or nitrogen offers no advantage in most of the cases because the vapours will absorb some of the gas and the deposits obtained will be contaminated. It is therefore desirable to evaporate in a vacuum not exceeding 10^{-3} mm. Hg preferably at a pressure of 10^{-5} mm. Hg. As the pressure is reduced the boiling point shifts closer to the melting point and as the temperature is raised, the vapour pressure rises quickly to values where very high evaporation speeds could be obtained but it is not in all cases advisable to operate at high evaporation speeds. For instance, when purifying metals, the temperature of the metal should be kept below its melting temperature for a while to facilitate the distillation of the more volatile contaminants. Also, in thin film preparation the temperature should be kept comparatively low in order to protect the substrate. In addition, slow evaporation facilitates a more accurate control of the growth of the deposit and reduces the risk of substantial evaporation of filament or crucible material. Values of the speed of evaporation for platinum are given in a table reproduced below.

Vapour Pressures and Evaporation Speeds of Platinum

Temperature °C	*Vapour Pressure mm. Hg*	*Speed of Evaporation g/cm²*
730	3.2×10^{-18}	8.3×10^{-20}
880	2.8×10^{-16}	1.5×10^{-17}
1030	1.4×10^{-13}	4.5×10^{-15}
1230	7.5×10^{-11}	5.5×10^{-12}
1480	2×10^{-7}	2.5×10^{-8}
1771 (m.p.)	1.4×10^{-4}	9.3×10^{-4}

Vol. 4 1954

III — Vacuum Processing Techniques — III
Contd.

Abstract No. and References

Platinum is typical of materials difficult to evaporate and the evaporation speeds of practically all other materials is higher by several orders of magnitude. The article concludes with the description of a small laboratory plant for the study of the evaporation of metals developed in Eastern Germany.

Sommaire : Discussion sur les lois fondamentales régissant l'évaporation des métaux dans toutes les gammes de pression.

Article by H. Laporte *Chem. Tech.* 5, Nov. 1953 632-634

38 — DISTILLATION — 38

Rate of Evaporation of Glycerol in High Vacuum 39/III

See Abstract No. : 39/I

Some Aspects of Molecular Distillation 40/III

O *United Kingdom.* Theory and practice of the technique of molecular distillation is reviewed. There are three principally different distillation techniques : Distillation by boiling, normal evaporative distillation and molecular (evaporative) distillation. The evaporation rate is governed by the number of molecules which escape from the liquid free surface and the distillation rate by the number of molecules which reach the condenser unhindered by collisions with molecules of the residual gas. In molecular distillation the values of the two rates should be close to each other because the distance ' d ' between evaporating and condensing surface is kept smaller than the mean free path of the residual gas at the (low) pressure applied. The length of the mean free path required can be expressed by :

$$\lambda_E = \frac{2.3 \times 10^{-20} \times T}{p_{mm} \times \sigma^2}$$

where T is the temperature in °K, p_{mm} the pressure in mm. Hg and σ the diameter of the molecule in cm. The approximate size of an evaporated high molecular-weight molecule is 8 Å. For a gap of 2 cm. the highest pressure permissible is 3×10^{-3} mm. Hg at 20°C. The effective evaporation rate is given by :

$$n_e = 5.83 \times 10^{-2} \times p_{mm} \sqrt{\frac{1}{M\,T}} \quad [\text{g.-mol./cm}^2\text{/sec.}]$$

where p_{mm} is the saturation vapour pressure at equilibrium temperature, T (°K) and M is the molecular weight and, for practical purposes to arrive at the distillation rate applicable, this expression is multiplied by a factor f which has normally a value of 0.5–1. The difference between evaporation and distillation rate is caused by the fact that collisions occur also between the evaporated molecules themselves. A full discussion of the theory of the probability of such collisions is given. The effect of temperature on the distillation of mixtures is examined and it is shown that in general the separation of a light component is best carried out at the lowest permissible temperature. Assuming that Raoult's law applies, α, the relative volatility of the two components can serve as a measure of the degree of separation obtainable and can be expressed by :

$$\alpha = \frac{p_1}{p_2} \sqrt{\frac{M_2}{M_1}}$$

O The author proceeds to discuss the various designs of molecular stills in existence : *Tray Still.* This still has a small capacity. The depth of the liquid in the trays must be kept at a minimum. *Circulating Batch Still.* A still of this type suitable for an initial charge of 350 c.c. is described and shown in an illustration. *Degasser and Devolatiliser.* This apparatus is employed to degas liquids and remove the lightest volatiles prior to the main molecular distillation treatment. *4-stage Continuous Still.* This still facilitates continuous operation and incorporates a degassing unit situated in front of the first stage. Separate gear pumps continuously extract the residue and the distillates from each of the four stages. *Centrifugal Still.* This still is especially suitable for the treatment of heat-sensitive materials. The quality of separation obtained in this type of still is not confirmed yet. Discussing the applications of molecular distillation the author states that re-evaporation from the condenser kept at 20°C is negligible only as long as the vapour pressure of the condensate does not exceed 10^{-5} mm. Hg at that temperature. Another limiting factor is the decomposition of organic substances which restricts the evaporation temperature to the range of 300–350°C. Generally speaking, the following materials can be successfully treated by the molecular distillation process : Petroleum oils, fish oils, triglyceride, soy bean, corn oils and miscellaneous acetates, fatty acids and esters.

Sommaire : Une revue de la théorie et de la pratique de distillation moléculaire.

Paper by G. Burrows *Spec. Print. Inst. Chem. Engrs.* 12.12.1953

A Three-Stage Molecular Still 41/III

South Africa. Falling-film molecular stills have the advantage of low working temperatures and short contact times but their separation efficiency is low. An all-Pyrex three-stage falling-film molecular still is described which is claimed to have a separation efficiency 2½ times (at least) that of a single-stage still. The still has a length of 73 cm. carrying at the top a reservoir of 170 c.c. volume. The three stages are situated vertically above each other. The distillate is collected in a pig receiver at the bottom of the column and the residues

III — Vacuum Processing Techniques — III
Contd.

Abstract No. and References

in a flask, positioned lower down, which serves at the same time as the receptacle for the distilland prior to operations. Before treatment is started the material is passed by means of an ' air lift ' pump to the reservoir at the top. From there it is fed through a drop counter on to the first stage, which like the other two consists of a piece of tubing positioned vertically and filled with a solvent heated by separate means in each case. The distilland flows over the outer (specially roughened) surface of this tube, the distillate being collected in a gutter (one to each stage) and the residue being collected on an inclined flange at the bottom of the tube. From here the residue passes into feeding tubes which convey it to the residue flask mentioned before. The distillate of each stage collects in a small flask, from where it is conveyed to the next stage. The operational pressure is 10^{-2} mm. Hg produced by a 3–stage oil diffusion pump unit. After completion of a run the residue collected in the flask is returned to the reservoir and another run is started. By way of example the treatment of commercially available oleic acid is described. The initial charge was 80 g. The temperature of the stages was 96°C. In the first run 25% of the distilland was removed in seven distillates. Then a second run was carried out after raising the stage temperature to 102°C and a fraction of 2–3 grams was collected every 1½ hours. At the end of the process, 0.6 g. of the distilland was found in the cold trap, 2.3 g. in the still and 2.0 g. in the ' air lift ' line and the reservoir.

Article by D. A. Sutton *Chem. & Industr.* 26.12.1953 1383-5

Sommaire : Une colonne de distillation moléculaire à 3 étages, du types à écoulement vertical de couches, a été développée pour des charges initiales de 80 g de matière; son efficacité est dite d'etre 2.5 fois plus grande que cette d'une colonne à un étage.

O

Apparatus for Vacuum Distillation 42/III

United States. In a molecular still the vapours of the diffusion pump liquid are introduced at the surface of the evaporating film and by entrainment of the evaporated molecules, aid in carrying them to the condenser. (*Chemical Abstracts*)

W. B. Whitney & Phillips Petroleum Co. *U.S. Pat.* 2,647,862

Sommaire : Une colonne de distillation moléculaire d'un modèle spéçial est décrite.

Vol. 4 195

39 — MISCELLANEOUS PROCESSES — 39

The Influence of Barometric Pressure on Watch and Chronometer Rates 43/III

See Abstract No. : 15/I

Design and Operating Technique of a Vacuum Drying Oven 44/III

See Abstract No. : 23/III

Pre-Cooling Fruit and Vegetables 45/III

United States. Details are given of two vacuum-cooling plants employed for the pre-cooling of fresh produce prior to shipment or cold storage. In a vacuum of 29.8 inch Hg water boils at 32°F and the evaporation of 1 lb. of water absorbs 1,000 B.t.u. of heat. The extraction of 1 lb. of water, from 100 lb. of lettuce at that pressure reduces the lettuce temperature by 10°F. In the case of the first plant described the temperature in the cooler is reduced to 32°F in 15 minutes and held there for 2 minutes. The final temperature of the produce was 34°F. The vacuum required is produced by steam ejectors. The second plant is made up from three cooling chambers which are operated in series in such a manner that each chamber is partially evacuated before the actual cooling operation starts. Here the temperature of the produce is reduced from 73°F to 32°F in 30 minutes but in order to cool the produce right through, the pressure is allowed to increase for a short period at a suitable moment during the process. The overall production time for one batch was 70 minutes. The first plant had a capacity of 50 crates (= 50 × 36 heads of lettuce, for instance) per charge. The second plant had a capacity of 72 crates per operation and was running at full load for eight months of the year. The process is successful in the case of leafy vegetables including Italian sprouting brocoli, celery, spinach, lettuce and cole slaw salad but strawberries, beans and peas in pod are found to burst, and tomatoes, bananas and cauliflowers are also found unsatisfactory. The quality of some varieties of apples deteriorates in the process. The loss in weight of pre-packed vegetables treated by this method was investigated and found to range from 2.2 to 3.2%. Vacuum-cooled produce can be packed and transported more efficiently and cheaply than produce refrigerated with ice. Vacuum cooling lengthens the shelf life of the material and avoids the risk of damage by bacterial activity and mould growth caused by the melting ice.

O

Article by Anon. *World Refrig.* **4,** July 1953 386-389

Sommaire : Des détails sont donnés sur deux appareils de congélation sous vide, employés dans l'industrie alimentaire en Amérique, pour la congélation à grande échelle de légumes et produits alimentaires variés.

The Recovery of Essence from Florida Orange Juices 46/III

United States. A system for recovering water-soluble essences of orange juice under vacuum at temperatures of 110–115°F is described. The orange juice was not noticeably deteriorated by processing at those temperatures. It was necessary to evaporate at least 20% of the juice to obtain the bulk of the essence. A relation existed

III — Vacuum Processing Techniques — III
Contd.

Abstract No. and References

between the aroma of the essence and the peel-oil content. The use of the water-soluble essence enhanced the floral character of the aroma of the freshly prepared concentrate and reconstituted product, but this aroma was lost in 6 months at 0°F.

(*Chemical Abstracts*)

Sommaire : Une méthode de récupération de l'essence pendant la production de jus d'orange, comprenant l'emploi d'un équipement à vide, est décrite.

Article by D. A. Morgan M. K. Veldhuis R. K. Eskew & G. W. M. Phillips *Food Technol.* **7**, 1953 332-336

New Volatile Flavour Process 47/III

United States. The process aims at retaining the original quality and flavour of fruit juices after concentration. The fresh fruit juice is passed through a falling-film evaporator maintained at 20 mm. Hg and 70°F. Some of the vapours flashing from the separator are condensed in the condenser of the evaporator held at 50°F. The remaining vapour passes over a second condenser, operated at 32°F where again a part of it is condensed, solidifying in the process. The rest of the vapour is condensed in a third liquid nitrogen-cooled condenser with a temperature of –285°F. When about 10% of the original charge has been evaporated the 2nd and 3rd condensers are put out of action and the condensate collected in both these condensers is added to the end product after completion of the concentration process.

Abstract by Anon *Food Engng.* **25**, Sept. 1953 196

O *Sommaire* : Un nouveau procédé visant à la conservation du parfum original du fruit dans la concentration des jus d'orange, employant trois condenseurs, est décrit.

Puffed Corn Production and Method of Making the Same 48/III

See Abstract No. : 26/III

Puffs Foods Continuously in Special Vacuum Process 49/III

See Abstract No. : 25/III

How They Put That Quality into Danish Canned Ham 50/III

Denmark. High quality ham is hygienically processed in a Danish plant by two different methods. The ham is first pressed into a can of selected size with a pneumatic press able to apply a 120 lb./sq. inch. thrust. The can is then transferred to a dip-soldering unit where a lid is fitted, clamped pneumatically, double-seamed and soldered. Ham canned in this way is passed on for treatment either by the ' Evacuating ' process or by the ' Supertri ' process. In the former method the can is placed in one of two airtight chambers. The chamber is evacuated by a small vacuum pump. A soldering iron fitted to the inside of the chamber is then manipulated from outside by the operator and used to seal the pumping hole in the can when the desired degree of vacuum (indicated by a dial gauge) has been reached. The canned ham is finally cooked in open vats, cooled and stored under low temperature. The ' Supertri ' system aims at producing a more durable ham. In this method, a rack carrying about 50 dip-soldered cans is rolled into a cabinet-type steam-cooker where a much higher cooking temperature is employed. Each can is then passed, while still warm, to a vacuum chamber where the juice is pressed out of the can during evacuation. The juice is drawn into a calibrated cylinder serving as a check on the progress of the operation. At the given moment, the can is sealed with a soldering iron in a similar way as before. After completion of processing the cans are cooled down and ready for shipment.

Article by Anon. *Food Engng.* **25**, Dec. 1953 127 & 203

O *Sommaire* : Deux différents procédés de fermeture sous vide de boîtes de conserve, contenant du jambon, sont rapportés.

Rust in Water System Checked by Deaeration 51/III

See Abstract No. : 10/I

The Solubility of Gases in Lubricating Oils and Fuels 52/III

See Abstract No. : 46/I

Cosmic-Ray Production of Helium in Meteorites and their Ages 53/III

United Kingdom. If a means of accurately assessing the ratio of the helium–3 to the helium–4 for helium produced by cosmic rays in iron meteorites could be found, the ages of these meteorites could be calculated with the help of the classical radio-activity method. For reasons stated, the ratio varies at different points inside the meteorites and one way of estimating its values is to determine upper and lower limit values. The highest observed ratio is 0.315 and in one large meteorite, ' Carbo ', its value varied from 0.288 at 3 cm. depth to 0.277 at 29 cm. depth. Also, the total helium content was found to decrease in depth. In order to obtain an estimate the measured values of $He^3/(He^3+He^4)$ inside ' Carbo ' were plotted against the calculated ratio of (helium due to primary radiation)/(helium due to primary radiation + helium due to secondary radiation)

III — Vacuum Processing Techniques — III

Contd.

Abstract No. and References

at the respective depths. This gave a linear relationship. By extrapolation to 100 and to 0% primary radiation, limiting values for the ratios in question of 0.4 and 0.25 respectively were obtained. This agreed roughly with the measurements in ' Carbo '. In the present work mainly meteorites with a (He^3/He^4) ratio less than 0.31 were examined. On the basis of the estimated ratios the helium–4 content could be calculated and from the uranium and thorium content the age could thus be estimated. The method of extracting the helium has been described previously (see also Vol. III, Abstract 111/I). The helium–3 and helium–4 was assayed in the mass spectrometer and the uranium and thorium, present to the extent of 10^{-8} g.p.g. meteorite, was analysed by a method described elsewhere. By this method, maxima and minima ages are estimated for a number of meteorites and range from about one million to 300 million years.

Sommaire : Une méthode de détermination de l'âge approximatif de météorites de fer est discutée, et certaines estimations d'âge, obtenues par cette méthode sont données.

Article by
J. C. Dalton
F. A. Paneth
P. Reasbeck
S. J. Thomson
& K. I. Mayne
Nature
172, 26.12.53
1168-1169

Note on the Preparation of Thin Vanadium Targets

54/III

United States. The preparation of targets in the form of thin films on Formvar backing by the evaporation technique is often difficult. The normal methods, *e.g.* evaporation from a carbon boat, are not successful in the case of materials such as vanadium, manganese and cobalt. A successful method of dealing with vanadium in this respect is reported. The vanadium pellet is held between the hollowed end of one and the pointed end of another carbon rod suspended horizontally and electric current is passed through the rods. As a result the pellet is fused to the female rod. When the fusion process is completed the rod carrying the vanadium pellet is ground to facilitate better isotropic evaporation and the second rod is ground to nearly filament point resting against the vanadium pellet during the actual evaporation process. Targets with an effective thickness of 5–50 kV for 6–Mev deuterons have been prepared in this manner and have withstood several thousand microcoulombs of bombardment with beam currents of 0.3 μA without deterioration.

Sommaire : Description d'une méthode pour préparer sans aucune difficulté, des cibles de vanadium, par le procédé d'évaporation sous vide.

Note by
J. E. Schwager &
L. A. Cox
Rev. Sci. Instrum.
24, Oct. 1953
986

Vol. 4 1954

IV — Special Subsidiary Subjects — IV

Abstract No. and References

40 — GASES AND VAPOURS — 40

The Liquid Helium Plant of the Physical Laboratory of the University of Louvain 1/IV

See Abstract No. : 35/II

Mass Spectrometric Determination of Gas Impurities Separable by Condensation 2/IV

United States. The mass spectrometric determination of condensable impurities in non-condensable gases is an established procedure and has been applied, for instance, to investigations of smog in the atmosphere and of the presence of condensable vapours in room air. The method is based on the combination of system volume and instrument sensitivity relating the amount of impurity traced to the size of the sample. The same method cannot be applied with equal success where it is a question of determining non-condensable impurities in condensable gases because the amount of gas trapped in the condensate is unknown. This difficulty can be overcome by introducing an internal standard of the same gas type as the impurities into the mixture in quantities which, on one hand, should permit the determination of the ratio of main component to internal standard from the mass spectrometer record and, on the other hand, should permit determination of the ratio of impurities to standard after condensation of the main component. The choice for an internal standard for the determination of non-condensable impurities should be such that solubility effects are minimised. Also, the choice of the standard is limited when liquid air or liquid nitrogen is used as the condensing medium as compared with conditions prevailing at higher condensation temperatures. In the present work the condensing medium and the internal standard used were liquid nitrogen and neon respectively. For making the mixtures of non-condensable impurities in condensable gas the minor components (about 30 to 200 parts per 100,000 parts of the main component) were introduced into a 500 ml. gas bottle evacuated to 10^{-5} mm. Hg through an inlet covered with a mercury sealed sintered disc. Calibrated gas pipettes were used for the purpose which ensured an accuracy of about 1%. Then the main component was admitted to the bottle *via* the vacuum line connecting with a cold storage bulb into which it had been condensed previously. The latter was warmed up for the purpose. Mixtures of condensable impurities in non-condensable gases were made in the gas bottle by introducing liquids through the sintered disc by means of special capillary dippers. Then the non-condensable component was added from a large storage reservoir as before. The gas mixtures were blended with the internal standard in a special mixing bulb of 100 ml. volume which formed part of the vacuum system and was filled with the respective permanent gas to a pressure of 8 to 10 mm. Hg after evacuation. Then, the stopcock controlling the line connecting the gas bottle and mixing bulb were opened for a short while and the pressure in both vessels was roughly equilibrated. The procedure in the case of condensable impurities was slightly different. The analysis in the mass spectrometer was carried out by conventional methods. The method of evaluating the results of the measurements is described in detail. The results for several synthetic mixtures are tabulated showing a difference between the mole % added and the mole % found not exceeding 0.03%. The method could be employed for the determination of much lower concentrations of impurities than those used in the experiments described here but there are practical limits such as the range of the mass spectrometer used and other factors, which are discussed in detail.

Sommaire : Présentation d'un méthode utilisant un gaz standard interne dans le spectrographe de masse pour la détermination de traces d'impurités non-condensables dans des gaz condensables

Article by A. S. Newton *Analyt. Chem.* 25, Nov. 1953 1746-1749

Rapid Interferometric Analysis of Germane-Hydrogen Mixtures 3/IV

United States. During electrolysis of solutions containing various germanium compounds the hydrogen evolved at the cathode contains a certain amount of germane (germanium hydride, GeH_4). The Rayleigh-Zeiss interferometer fitted with a 500 mm. double-chamber gas cell can be used for the analysis of these mixtures. In this method pure hydrogen is filled into one chamber and the mixture into the other and the difference of refractive index between both is determined. This is achieved by splitting the light of a tungsten lamp into two separate beams each passing through one of the two gas chambers and through two identical glass plates, recombining on emergence to form interference fringes. A difference of refractive index is indicated by a sideways displacement of the band system. This is compensated and measured by adjusting the inclination of one glass plate. The exact point of compensation can be determined by comparing the first band system with a second system where the two beams have travelled identical paths. The method requires initial calibration with germane-hydrogen mixtures of known proportions. Details are given of the method of calibration involving the use of 15 different mixtures ranging from 0.64 to 12.58 mole % germane content. An illustration of the gas handling system is given. It consists essentially of a U-tube normally filled with mercury, one limb of which is graduated and can be connected either to a hydrogen or a germane reservoir. After admission of one of the gases to the graduated limb the mercury level in both limbs was levelled up and the non-graduated limb exhausted. In this way volume and pressure of each gas could be determined and the composition of the mixture calculated. Similar details of the normal analytical procedure are given. The method is accurate within $\pm$ 0.059 mole % germane, covers a range of 0 to 15 % germane and requires about 10 minutes for one determination.

Sommaire : Des mélanges d'hydrogène et de germane, tels que ceux émanants de l'electrolyse de solutions contenant différents composés de germanium, peuvent être analisés dans un interferomètre Raileigh-Zeiss équipé d'une cellule à gaz à double chambre. Des détails sont donnés sur cette méthode.

Article by M. Green & P. H. Robinson *Analyt. Chem.* 25, Dec. 1953 1913-1914

IV — Special Subsidiary Subjects — IV
Contd.

Abstract No. and References

Rotary Vacuum Reaction Chambers

4/IV

United Kingdom. An apparatus is described for treating powdered solids in quantities of the order of 1 kg. by de-gassing it, reacting it with a gas and then subsequently removing samples for analysis while the bulk of the powder remains in vacuum. The apparatus is particularly suitable for the study of the sorption of gases or vapours on solids in cases, where estimation by pressure changes is unreliable and the use of a quartz fibre sorption balance is prohibited because the gases used in the experiments are corrosive. It further facilitates the study of the mixed sorption of two or more sorbates on the same sorbent independent of pressure changes and the preparation of substances under controlled vacuum or gaseous atmospheres. Two different designs of reaction chambers, both cylindrical (axis horizontal) in shape, are described. In the first, the powder is stirred by a six-bladed paddle revolving at about 6 r.p.m. inside the chamber. Samples for analysing are transferred from the chamber *via* a separately pumped vacuum lock to atmosphere or to an auxiliary vacuum system. The lock is joined to the chamber at the lowest point and is normally shut off by a plunger-type valve fitted with a pumped Wilson seal so that when the valve is opened powder falls into the lock. The quantity of powder withdrawn from the reaction chamber can be controlled visually by means of an observation window inserted in the lock chamber. Certain difficulties were experienced with this model, for instance :—The close fit between the paddle and the chamber makes the former prone to jamming, in certain instances abrasion due to powder collecting between the rubbing surfaces results in contamination of the powder and finally, the surface area of soft powders may be increased. These difficulties were overcome in the second design, referred to as the 'cement mixer' model, by enclosing the powder in a drum which has a sliding cover. Apertures in the curved surface of both drum and cover may be separated or made to coincide by displacing the drum spindle. The gas inlet tube passes through the centre of the cover-plate and through a central hole in the face of the drum. Both models described were made of copper but harder materials such as stainless steel are preferable, where a danger of abrasion by the powder exists.

Sommaire : Description est faite de chambres de réaction sous vide pour l'étude du phénomène de sorption de solides (poudre) où il n'est pas practique de mesurer les changements de pressions.

Article by J. F. Duncan & D. T. Warren *J. Sci. Instrum.* **30**, Dec. 1953 462-464

41 — FREEZE DRYING — 41

Study of the Factors Governing the Rate of Freeze Drying of Blood Plasma

5/IV

See Abstract No. : 42/I

Some Considerations Relating to the Design of Freeze Drying Plant

6/IV

United Kingdom. Existing large-scale freeze-drying plant can be classified into three groups : distal, medial and proximal, according to the relative position of the evaporating and condensing surfaces in the plant. In the distal type the refrigerated condenser is a unit separate from the drying chamber and connected to the drying chamber by wide-bore pumping lines. In this case the pumps have to deal with a mixture of permanent gases and large volumes of water vapour. In the medial type, the evaporating and condensing surfaces are sufficiently close to each other for the condenser to act as a pump with respect to the water vapour and to facilitate the use of smaller vacuum pumps dealing with the permanent gases only. Typical of this type is the plasma drying plant designed by Greaves in 1946 which employs a coiled pipe condenser. Due to the particular operating conditions in that plant it was found that ice forms on the condenser in such a manner that only a small proportion of the total coil surface is actually available for heat transfer. The author, in association with other workers, developed a new design of plasma drying plant which may be classified as of the proximal type and is aimed at improving the performance of the condenser. The drying chamber is a cylinder, positioned vertically. Its height is 3 ft. 6 inch. and its diameter is 3 ft. The operational position of the drying head is in the centre. Two condensers are used, facing each other and positioned between the drying head and the chamber wall. Each condenser consists of a single layer of tubes, closely packed, to cover a rectangular area of 3 ft. by 2 ft. The initial clearance between the bottles and the condensers is 3½ inch. The condensers are operated with Freon 22 and have an initial temperature of –45 °C which falls to –60 °C towards the end of the 48 hour drying period. The drying head holds 154 bottles, containing 60 l. of plasma. The total heat input to the drying head is 1,250 W. The pumping unit consists of a small diffusion pump backed by a single-stage rotary pump. Baffles are provided to prevent water vapour from reaching the pump. The pressure during the equilibrium period is 0.15 mm. Hg and falls to 0.03 mm. Hg at the end of the drying operation. Ice forms on the inward surfaces of the condensers only and can be quickly removed in the form of a sheet by blowing air into the chamber. Reference is made to publications by Ede which appear to justify the principal points of the design described, *i.e.* low operational pressure and a small distance between the evaporating and condensing surfaces. But the latter cannot be decreased below a certain value in order to avoid substantial heat losses by radiation. In the present plant such losses during equilibrium drying period amount to about 4 % of the total heat input.

Sommaire : Des détails sont donnés sur un appareil à lyophiliser le plasma dans lequel le condenseur et la surface évaporante sont très prêts l'un de l'autre et où le minimum de glace se forme durant l'opération.

Paper by R. A. Kekwick *Preprint Inst. Refrig.* Nov. 1953 4 pp.

IV — Special Subsidiary Subjects — IV

Contd.

Abstract No. and References

A Simple Laboratory Apparatus for Freeze Drying

7/IV

Germany. There is no small freeze drying plant available commercially which would be of use to institutions and laboratories which require such apparatus only occasionally for the preparation of sera, virus cultures, etc. The author describes a device built for the purpose which has been employed with success intermittently for the drying of sera over a period of two years. The material to be processed is fed into ampoules. The depth of the material in each ampoule is about 1 cm. Some 30 ampoules are placed in a freezing cabinet and their contents are frozen to –10°. Subsequently the ampoules are removed into a desiccator of a special design which has a diamenter of 25 cm. At the bottom the desiccator carries two trays, situated above each other, filled with phosphorus pentoxide. A charge of about 1,000 g. of phosphorus pentoxide should be used for each 100 cm³ of processed material. Above the two trays is a perforated plate made from porcelain, which serves as the support for the ampoules. At the top, the desiccator has a stop-cock-controlled outlet to the pumping unit. The latter consists of a single-stage glass mercury pump backed by a single-stage rotary pump. In this arrangement 100 cm³ of material can be dried satisfactorily in 12 hours, operating at a pressure of 0.1 mm. Hg. The greater part of the moisture withdrawn from the material is absorbed by the phosphorus pentoxide in the two trays mentioned. Any traces of water vapour entrained in the air entering the pumping line is absorbed by a small amount of phosphorus pentoxide in a paper boat inserted in the pumping line. During the absorption of moisture by the phosphorus pentoxide in the two trays a certain amount of heat is developed which is radiated to the processed material thus accelerating the drying process.

Note by K. E. Scheer *Zentralblatt* 165, March 1953 311-313

Sommaire : Description d'un petit appareil de lyophilisation pour le laboratoire qui nécessite seulement pour son fonctionnement l'action deséchante du P_2O_5 et une pression de 0.1 mm. Hg.

Method of Freeze Drying of Histological Preparations

8/IV

Sweden. A method of freeze drying histological preparations is described which avoids the use of high vacua and of a condenser. The preparation having a thickness of 1–2 mm. is placed in a small cylindrical wire-net container and frozen to –160°C in isopentane. The frozen preparation in the container is placed in a glass receptacle which holds a small amount of frozen paraffin at the bottom. The upper end of the glass receptacle is connected to a rotary vacuum pump. Air can be admitted to the glass vessel through an air drying train consisting of a silica gel column, a receptacle filled with concentrated sulphuric acid and a column of dehydrite. Finally, the air passes through a liquid air trap and a spiral glass coil which joins the glass receptacle near the bottom. The latter coil and the glass receptacle are immersed in a Dewar bottle filled with alcohol and solid CO_2. In operation, the pump draws air through the whole assembly at a rate of 3 l./h., measured at atmospheric pressure. When the air passes through the spiral coil its temperature is reduced to that of the sample, approximately –30°C, because the coil and the sample are immersed in the same freezing solution. The pressure in the drying receptacle is maintained at 5–10 mm. Hg. The air circulation is controlled by means of stopcocks. After about 6–10 hours the pump is shut off, the stopcocks are closed and the paraffin at the bottom of the drying receptacle is melted. As a result, the preparation sinks into the paraffin and can be safely removed for final embedding. Where the preparation is sensitive to oxidation, nitrogen instead of air should be circulated.

Note by L. Treffenberg *Arkiv. Zoologi* 4, No. 12, 1953 295-296

Sommaire : Description est faite d'une méthode de lyophilisation de préparations histologiques évitant l'emploi d'un vide poussé et de condenseurs.

The Preservation of Arterial Grafts by Freeze Drying

9/IV

See Abstract No. : 40/I

Spare Parts for Humans

10/IV

See Abstract No. : 16/I

45 — ELECTRONICS — 45

High Temperature Materials for Vacuum Service

11/IV

See Abstract No. : 21/II

Discharge Mechanisms in Cold-Cathode Valves

12/IV

United Kingdom. A general introduction is given to discharge phenomena in cold-cathode valves. If a glass envelope fitted with plate electrodes, 1 or 2 cm. apart and supplied with d.c. is filled with a few millimetres Hg of monatomic gas and if a source of free electrons in the form of radio-active material, for instance, is provided, the electrons thus generated will be attracted to the anode in the vessel. As long as the potential difference between the electrode is small, the electrons on their way to the anode will collide with the gas atoms elastically. If the potential difference between the electrodes is raised the energy of the electrons is greater and the collisions will cause excitation of the atoms. The latter will return to their normal state after 10^{-8} seconds and emit light in the process. In some cases meta-stable states of the atom will be created and the ground

IV — Special Subsidiary Subjects — IV
Contd.

Abstract No. and References

state will only be reached after collision with another molecule. Increasing the potential difference still further the electrons will begin to ionise the gas atoms and will start the motion of positive ions to the cathode. This results in the creation of additional electrons which amplify the initial stream to the anode. At this stage the so-called electron avalanche is created and the prevailing electron current density is expressed by :

$$j = j_0 \exp [\eta (V - V_0)] \quad \text{for } V \geq V_0 \qquad (1)$$

where j_0 is the initial electron current density, V_0 the ionisation potential of the gas and η the ionisation coefficient, defined as the number of ion/electron pairs produced by 1 electron in passing through a potential difference of 1 V. This coefficient varies with the electric field strength and the gas pressure and has a characteristic value for each gas. High η values can be obtained artificially by mixing the gas in the vessel with another of a lower ionisation potential than the meta-stable level potential of the former. Then the probability exists that meta-stable atoms of the one gas may ionise neutral atoms of the other, resulting in the generation of extra electrons. If the voltage is raised still further a self-sustaining discharge is obtained which is based on the generation of secondary electrons, to a minor extent due to positive ions colliding with atoms in the gas column, and to a larger extent resulting from the bombardment of the cathode by positive ions and meta-stable atoms. This state is expressed by :

$$j = j_0 \frac{\exp [\eta (V - V_0)]}{1 - \gamma (\exp [\eta (V - V_0)] - 1)} \qquad (2)$$

Where γ is the secondary ionisation coefficient defined as the number of secondary electrons produced at the cathode per ion reaching the cathode. Finally as V increases the state is approached where the denominator of (2) becomes zero. This value of V is V_B, the breakdown potential, which is defined as the voltage at which a gas between 2 electrodes allows a self-sustaining current to pass. In this connection Paschen's Law :

$$\frac{E}{p} = \frac{V_B}{pd}$$

is discussed which states that V_B is also a function of the pressure and the distance of the electrodes. In a graph, Paschen curves for various cathode materials are shown indicating that low-function materials have lower breakdown values. The author continues to discuss formative and statistical lags and space charge phenomena. Space charges are becoming evident only in current ranges above those of the Townsend discharge, *i.e.*, above 1μA. The positive space charge develops near the cathode as a result of the comparatively slow movement of the ions. The field strength increases and the potential applied can be reduced. A glow discharge develops on the cathode surface which has the characteristics of a constant low voltage over a wide range of current. Advantage is taken of this condition in the design of voltage stabilisers and voltage reference tubes. Once the glow discharge has covered the whole of the cathode, the current density and the glow voltage rise and arc discharge is initiated. The author proceeds to discuss certain types of cold-cathode tubes such as the cold-cathode triode (trigger) tube. The trigger cathode gap has a very low pd value requiring a comparatively low breakdown voltage. But once the main gap has broken down, the trigger electrode loses control over the main gap and the main discharge can only be extinguished by interrupting the supply. Similar details are given on cold-cathode tetrodes and multi-electrode tubes. A scale-of-ten counter tube is described which has the size of an ordinary radio valve.

Article by
R. W. Murray
Proc. Inst. Electronics
4th Quarter
1953
6–17

Sommaire : Les principes fondamentaux des phénomènes de décharge dans les tubes thermioniques sont discutés, et des détails sont données sur les tubes à cathode froide.

The Field-Emission Initiated Vacuum Arc. I. Experiments on Arc Initiation

13/IV

United States. It is known that electrical breakdown between metal electrodes can be initiated by field emission. The present work concerns a further study of that initiation process under conditions of excellent vacuum and a clean cathode surface. As the field current density from the single-crystal tungsten emitter is continuously increased, the normal emission is terminated by an explosive vacuum arc. Since this breakdown occurs in less than a microsecond, the experimental observations were obtained by use of pulse electronic techniques. The magnitude of the electric field, current density, and work function at the cathode were simultaneously determined prior to breakdown. From this investigation it has been shown that : (1) The vacuum arc was initiated at a critical value of the field current density of the order of 10^8 A/cm²; (2) breakdown was predictable and not random; in fact easily recognisable conditions preceding arc formation have been established; at current densities just below the critical value, an electron emission process was observed, which apparently involved both high electric fields and high temperatures; (3) arc formation did not require cathode bombardment by material from the anode or from residual gases; (4) breakdown was independent of the applied microsecond voltage in the range $5 < V < 60$kV, provided the critical current density was not exceeded; (5) the current during arc exceeded the initiating field current by a factor of at least 100.

(*Nuclear Science Abstracts*)

Article by
W. P. Dyke
J. K. Trolan
E. E. Martin
J. P. Barbour
Phys. Rev.
91, 1.9.1953
1043-1054

Sommaire : La formation de l'arc entre deux électrodes métalliques peut etre causé par un champ d'émission électronique entre ces deux électrodes.

The Field-Emission Initiated Vacuum Arc. II. The Resistively Heated Emitter

14/IV

United States. Electrical breakdown between clean metal electrodes in high vacuum was observed when the field current density at the single-crystal tungsten cathode exceeded a critical value of the order of 10^8 A/cm². At current densities just below the critical value, an electron-emission process was observed which apparently

IV — Special Subsidiary Subjects — IV
Contd

Abstract No. and References

involved both high temperature and high electric field. Calculations are presented for the emitter temperature increase due to the resistive mechanism for both the steady state and the transient solution. Emitter geometries used for the calculations approximated those obtained from electron micrographs of several emitters. The calculations show that the resistive heating was sufficient to melt the emitter at the critical current density, assuming the accepted value of the physical constants for the polycrystalline metal.

(*Nuclear Science Abstracts*)

Article by W. W. Dolan W. P. Dyke J. K. Trolan *Phys. Rev.* 91, 1.9.1953 1054-1057

Sommaire : La formation d'arc entre deux électrodes métalliques fut observée lorsque la densité du courant du champ sur la cathode de tungstène monocrystalline dépasse une certaine valeur. La relation entre l'émission électronique et la temperature de la cathode de tungstène a été recherchée.

Proton Synchrotron of the University of Birmingham

10/IV

United Kingdom. The proton synchrotron at Birmingham can now accelerate particles to 1,000 MeV. The Cosmotron at Brookhaven has an accelerating power of 2,300 MeV and a third, somewhat larger proton synchrotron is being built at Berkeley, California. These three machines serve for investigations in the field of nuclear physics in energy ranges above those for which synchro-cyclotrons have been designed. Some details of the Birmingham machine are given. The fully accelerated particles move in an orbit of 900 cm. diameter. The vacuum chamber is fitted into the C-shaped laminations of the circular magnet. The gap of the C is 21 cm. high. The vacuum chamber consists of a series of porcelain sections and is exhausted by five diffusion pumps each of a speed of 2,000 l./sec. and fitted with a water-cooled baffle. The operational pressure is 8×10^{-7} mm. Hg. The magnet is supplied with 1,100 V and produces a field of 12,500 Gauss. The protons are injected into the vacuum chamber tangentially from a Cockcroft-Walton H.T. generator. Their initial energy is 460 keV. Each particle receives an increment of energy equal to 200 eV per revolution which is just sufficient to maintain it in the mean orbit as the magnetic field rises. The respective electrode is operated with a radio-frequency voltage which is synchronised with the speed of the circulating protons. Full acceleration is obtained after 1 second. By that time the protons have made 5×10^5 revolutions. A fully accelerated pulse contains about 10^8 protons.

Article by Anon. *Nature* 172, 17.10., 1953 704-706

Sommaire : Détails sont donnés d'un synchrotron à protons de l'Université de Birmingham qui peut accélérer les protons jusqu'à environ 1,000 MeV.

A Mass Spectrometer for Leak Detection

10/IV

See Abstract No. : 33/II

47 — GLASS PROCESSING — 47

Metal-to-Glass Seals for Sub-λ Point Helium Apparatus

12/IV

United States. It is known that metal-to-glass seals incorporated in low-temperature vacuum apparatus which is exposed to superfluid helium give trouble because helium II leaks through the seals. On the other hand, metal-to-glass seals offer many advantages in the design and operation of such apparatus. The trouble caused by the seals can be eliminated if the leak rate can be reduced so that the partial helium pressure building up in the apparatus is kept below the operational vacuum requirements in the apparatus. This can be achieved by providing suitable pumping facilities and by elaboration of the conventional glass-to-metal seal design. For instance, in the particular case of the measurement of electronic heat capacities of metals below the helium λ point, the pressure in the calorimeter had to be maintained at 10^{-8} mm. Hg and gas evolution due to leakage and degassing had to be kept below 10^{-4} micron-litre/sec. For use with this apparatus the authors designed metal-to-glass seals for tubes of about 1 inch diameter and for wires of about 0.025 inch diameter which gave maximum leakage rates for superfluid helium very much lower than allowed for. The materials required for such seals are a hard glass withstanding thermal shock and a metal with poor magnetic properties at the low operating temperatures. The latter should either possess a coefficient of expansion matching that of the glass used or be of the low-strength highly ductile type employed in Housekeeper seals. The authors describe special tube seals, (*a*) of the copper Housekeeper type, where O.F.H.C. copper of a wall thickness of 0.003 inch was sealed to Corning 7720 glass after heat treatment for 3 hours at 950°C and a pressure of less than 10^{-5} mm. Hg, and (*b*) zirconium seals made from the pure metal with a wall thickness of 1/32 inch sealed to Corning 7052 glass. Oxidation of the metal was kept at a minimum. Similar information is given on special wire seals made from tungsten, copper and zirconium. Testing seals of this kind in a helium mass-spectrometer leak detector with a sensitivity of 5×10^{-9} micron-litre/sec. and exposing the seals alternatingly to room and liquid helium temperatures, produced no measurable leakage. An illustration is shown of a combined tube and wire seal constructed in accordance with the principles described for use in the experimental calorimeter work mentioned above.

Note by W. S. Corak & A. Wexler *Res. Nat. Bur. Stds.* 52, Oct., 1953 225

Sommaire : Description est donnée de modèles spéciaux de joints verre-métal sous forme de tubes ou fils qui facilitent leur emploi dans des appareils à vide fonctionnant à basse temperatures sans risque de fuites appréciables dues au hélium II.

Vol. 4 1954

IV — Special Subsidiary Subjects — IV
Contd

Abstract No. and References

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Article by
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W. P. Dyke
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Phys. Rev.
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15/IV

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Article by
Anon.
Nature
172, 17.10., 1953
704-706

A Mass Spectrometer for Leak Detection

16/IV

See Abstract No. : 33/II

47 — GLASS PROCESSING — 47

Metal-to-Glass Seals for Sub-λ Point Helium Apparatus

17/IV

United States. It is known that metal-to-glass seals incorporated in low-temperature vacuum apparatus which is exposed to superfluid helium give trouble because helium II leaks through the seals. On the other hand, metal-to-glass seals offer many advantages in the design and operation of such apparatus. The trouble caused by the seals can be eliminated if the leak rate can be reduced so that the partial helium pressure building up in the apparatus is kept below the operational vacuum requirements in the apparatus. This can be achieved by providing suitable pumping facilities and by elaboration of the conventional glass-to-metal seal design. For instance, in the particular case of the measurement of electronic heat capacities of metals below the helium λ point, the pressure in the calorimeter had to be maintained at 10^{-8} mm. Hg and gas evolution due to leakage and degassing had to be kept below 10^{-4} micron-litre/sec. For use with this apparatus the authors designed metal-to-glass seals for tubes of about 1 inch diameter and for wires of about 0.025 inch diameter which gave maximum leakage rates for superfluid helium very much lower than allowed for. The materials required for such seals are a hard glass withstanding thermal shock and a metal with poor magnetic properties at the low operating temperatures. The latter should either possess a coefficient of expansion matching that of the glass used or be of the low-strength highly ductile type employed in Houskeeper seals. The authors describe special tube seals, (*a*) of the copper Houskeeper type, where O.F.H.C. copper of a wall thickness of 0.003 inch was sealed to Corning 7720 glass after heat treatment for 3 hours at 950°C and a pressure of less than 10^{-5} mm. Hg, and (*b*) zirconium seals made from the pure metal with a wall thickness of 1/32 inch sealed to Corning 7052 glass. Oxidation of the metal was kept at a minimum. Similar information is given on special wire seals made from tungsten, copper and zirconium. Testing seals of this kind in a helium mass-spectrometer leak detector with a sensitivity of 5×10^{-9} micron-litre/sec. and exposing the seals alternatingly to room and liquid helium temperatures, produced no measurable leakage. An illustration is shown of a combined tube and wire seal constructed in accordance with the principles described for use in the experimental calorimeter work mentioned above.

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Note by
W. S. Corak &
A. Wexler
Rev. Sci. Instrum.
24, Oct. 1953
994

Cumulative Author Index, Vol. IV (1954)

Author	Title of Publication	Abstr. No.
Alpert, D.	Copper Isolation Trap for Vacuum Systems.	27/II
Anon.	Moisture Removal.	8/I
Anon.	Proton Synchrotron of the University of Birmingham.	15/IV
Anon.	Pre-Cooling Fruit and Vegetables.	45/III
Anon.	New Volatile Flavour Process.	47/III
Anon.	How They Put That Quality into Danish Canned Ham.	50/III
Anon.	Metal Melting Under Vacuum. Some Mechanical Problems Involved.	54/I
Baldwin, R. R., *et al.*	The Solubility of Gases in Lubricating Oils and Fuels.	46/I
Bancroft, G. H.	Metal Finishing by High Vacuum Metallisation.	2/III
Barbour, J. P., *et al.*	The Field-Emission Initiated Vacuum Arc. I. Experiments on Arc Initiation.	13/IV
Baxter, I. G.	A Differential Capacitance Manometer.	9/II
Behrent, L. F.	Fabrication of Radio-Frequency Micropotentiometer Resistance Elements.	22/I
Bigg, P. H., et al.	*The Influence of Barometric Pressure on Watch and Chronometer Rates.*	*15/I*
Bode, D. E., *et al.*	Photoconductivity of Indium Selenide.	31/I
Brady, J., *et. al.*	Vacuum Fusion Analysis.	37/III
Brown, A. R. G., *et. al.*	Density of Deposited Carbon.	24/II
Bruce, E. W., *et al.*	A Study of the Factors Governing the Rate of Freeze Drying of Blood Plasma.	42/I
Bryant, P. J., *et. al.*	Structure of Evaporated Films as a Function of Film Thickness.	11/III
Buckingham, J. D., *et al.*	Improvements i.o.r.t. Apparatus for Measuring Gas Pressure.	10/II
Burridge, J. C., *et al.*	Reflectivity of Thin Aluminium Films and their Use in Interferometry.	29/I
Burrows, G.	Some Aspects of Molecular Distillation.	40/III
Burrows, G.	Improvements r.t. the Sealing of Joints in High Vacuum and the Like Systems.	30/II
Carbide & Carbon Chemicals Co., *et al.*	A Metal Vacuum Lock for a Mass Spectrometer.	13/II
Carman, C. R., *et al.*	Puffed Corn Production and Method of Making the Same.	26/III
Cassignol, C., *et al.*	A Mass Spectrometer for Leak Detection.	33/II
Cobbold, A., *et al.*	Fine Structure in Polyethylene Terephthalate Fibres.	23/II
Columbe, M. J., *et al.*	An Improved Electron Diffraction Instrument.	17/II
Corak, W. S., *et al.*	Metal-to-Glass Seals for Sub-λPoint Helium Apparatus.	17/IV
Cox, L. A., *et al.*	Note on the Preparation of Thin Vanadium Targets.	54/III
Crittenden, E. C., Jr., *et al.*	Thin Films of Ferromagnetic Materials.	9/III
Cross, I.	Vacuum Metallising and Plating—A Comparison.	4/III
Dalton, J. C., *et al.*	Cosmic-Ray Production of Helium in Meteorites and Their Ages.	53/III
Daniel, S. G., *et al.*	The Solubility of Gases in Lubricating Oils and Fuels.	46/I
Daubeny, R. de P., *et al.*	Fine Structure in Polyethylene Terephthalate Fibres.	23/II
Davey, T. R. A.	Vacuum Dezincing of Desilverised Lead Bullion.	33/III
DeLollis, N. J.	Method of Preparing Nickel Powder with a Large Surface Area.	57/I
DeMaria, V. C.	A Portable High Vacuum System.	1/I
Deutsch, K., *et al.*	Fine Structure in Polyethene Terephthalate Fibres.	23/II
Dolan, W. W., *et al.*	The Field-Emission Initiated Vacuum Arc. II. The Resistively Heated Emitter.	14/IV
Duncan, J. F., *et al.*	Rotary Vacuum Reaction Chambers.	4/IV
Dyke, W. P., *et al.*	The Field-Emission Initiated Vacuum Arc. I. Experiments on Arc Initiation.	13/IV
Dyke, W. P., *et al.*	The Field-Emission Initiated Vacuum Arc. II. The Resistively Heated Emitter.	14/IV
Edwards High Vacuum Ltd., *et al.*	Improvements i.o.r.t. Vapour Vacuum Pumps.	5/II
Elliott, K. W. T., *et al.*	The Influence of Barometric Pressure on Watch and Chronometer Rates.	15/I
Eskew, R. K., *et al.*	The Recovery of Essence from Florida Orange Juices.	46/III
Evans, E. C., *et al.*	Modified Hoke Connector.	31/II
Fairbrother, F., Jr., *et al.*	Sputtering of Stainless Steel by Protons in the 30-80 keV Range.	21/III
Fast, J. D.	The Production of Pure and Deliberately Contaminated Iron. Experimental Investigations into the Properties of these Metals.	34/III

Vol. 4 1954

Cumulative Author Index, Vol. IV (1954)

Contd.

Author	Title of Publication	Abstr. No.
Finch, G. I., *et. al.*	The Diffraction of 150 kV Electrons.	16/II
Foster, J. S., Jr., *et al.*	Sputtering of Stainless Steel by Protons in 30-80 keV Range.	21/III
Fourie, D. J.	The Photoelectric Effect of Thin Bismuth Films.	20/III
Frank, W.	Oil Diffusion Pump in Glass with a Novel Fractionating Chimney.	4/II
Fultz., C. R., *et al.*	A Metal Vacuum Lock for a Mass Spectrometer.	13/II
Gardiner, S. D.	Design and Operating Technique of a Vacuum Drying Oven. Part 1. Design of the Oven.	23/III
Geller, R., *et al.*	A Mass Spectrometer for Leak Detection.	33/II
General Electric Co., Ltd. *et al.*	Improvements i.o.r.t. Apparatus for Measuring Gas Pressure.	10/II
Goetz, G. A.	Vacuum Process Brightens Plating Picture.	3/III
Green, M., *et al.*	Rapid Interferometric Analysis of Germane-Hydrogen Mixtures.	3/IV
Greve, L., de., *et al.*	The Liquid Helium Plant of the Physical Laboratory of the University of Louvain.	35/II
Guardite Corp., *et al.*	Puffed Corn Production and Method of Making the Same.	26/III
Haardick, H.	The Behaviour of Light-Sensitive Evaporated Layers in the Electron Microscope.	19/II
Haefer, R.	On the Mechanism of the Initiation of an Electrical Gaseous Discharge under the Action of a Transverse Magnetic Field in the Pressure Range 10^{-1} to 10^{-8} mm. Hg.	18/I
Hall, A. R., *et al.*	Density of Deposited Carbon.	24/II
Heidenhain, J.	Photographic Copying Process.	62/I
Hoffman, R. W., *et al.*	Thin Films of Ferromagnetic Materials.	9/III
Horton, W. S., *et al.*	Vacuum Fusion Analysis.	37/III
Hughes, H.	Use of P.T.F.E. in Impregnating Porous Materials.	22/II
Hull, L. W.	Pick a Vacuum System for Your Job.	6/I
Itterbeek, A., Van, *et al.*	The Liquid Helium Plant of the Physical Laboratory of the University of Louvain.	35/II
Kearsey, H. A., *et al.*	A Study of the Factors Governing the Rate of Freeze Drying of Blood Plasma.	42/I
Kekwick, R. A.	Some Consideration Relating to the Design of Freeze Drying Plant.	6/IV
Kuhn, H., *et al.*	Reflectivity of Thin Aluminium Film and their Use in Interferometry.	29/I
Lakhanpal, M. L., *et al.*	Boiling Point of Capillary-Condensed Water.	38/I
Langdon, A., *et al.*	Modified Hoke Connector.	31/II
Laporte, H.	The Evaporation of Metals and Metalloids in Vacuum.	38/III
Lecomte, J., *et al.*	A Few Remarks on the Electronic Circuit of a Leak Detector.	23/I
Le Grice Burnett, R., *et al.*	Improvements i.o.r.t. the Manufacture of Calcium.	36/III
Levinstein, H., *et al.*	Photoconductivity of Indium Selenide.	31/I
Lewis, H. C., *et al.*	The Diffraction of 150 kV Electrons.	16/II
Markey, P., *et al.*	Fine Structure in Polyethylene Terephthalate Fibres.	23/II
Marmo, F. F.	Absorption Coefficients of Nitrogen Oxide in Vacuum Ultra-Violet.	34/I
Martin, E. E., *et al.*	The Field-Emission Initiated Vacuum Arc. I. Experiments on Arc Initiation.	13/IV
Mayne, K. I., *et al.*	Cosmic-Ray Production of Helium in Meteorites and Their Ages.	53/III
Metropolitan-Vickers Electrical Co. Ltd., *et al.*	Improvements r.t. the Sealing of Joints in High Vacuum and the Like Systems.	30/II
Michaelson, H. B.	High Temperature Materials for Vacuum Service.	21/II
Miller, I. C.	Puffs Foods Continuously in Special Vacuum Process.	25/III
Ministry of Supply, *et al.*	Improvements i.o.r.t. the Manufacture of Calcium.	36/III
Moore, J. H.	Development of Commercial Vacuum Furnaces for Melting Metals and Alloys.	14/I
Moreau, J., *et al.*	A Mass Spectrometer for Leak Detection.	33/II
Morgan, D. A., *et al.*	The Recovery of Essence from Florida Orange Juices.	46/III
Murbach, H. P., *et al.*	The Origin of Stress in Metal Layers Condensed from the Vapour in High Vacuum.	7/III
Murray, R. W.	Discharge Mechanisms in Cold-Cathode Valves.	12/IV
Myncke, H., *et al.*	The Liquid Helium Plant of the Physical Laboratory of the University of Louvain.	35/II

Vol 4 195

Cumulative Author Index, Vol. IV (1954)

Contd.

Author	Title of Publication	Abstr. No.
National Lead Co., *et al.*	Production of Titanium Metal.	35/III
Newberry, S. P., *et al.*	An Improved Electron Diffraction Instrument.	17/II
Newton, A. S.	Mass Spectrometric Determination of Gas Impurities Separable by Condensation.	2/IV
O'Hara, W. W., *et al.*	Techniques for Measuring and Controlling Vacuum.	3/I
Owens, E. A.	A Rotating Anode Gas X-Ray Tube.	15/II
Paige, E.	A Metal High Vacuum Manifold.	26/I
Paneth, F. A., *et al.*	Cosmic-Ray Production of Helium in Meteorites and Their Ages.	53/III
Pate, J. W., *et al.*	Electrical Potential Differences Across the Normal Aorta and Aortic Grafts of Dogs.	41/I
Pery, A., *et. al.*	Reflectivity of Thin Aluminium Films and Their Use in Interferometry.	29/I
Phillips, G. W. M., *et al.*	The Recovery of Essence from Florida Orange Juices.	46/III
Phillips Petroleum Co., *et al.*	Apparatus for Vacuum Distillation.	42/III
Powell, A. R.	Very Pure Metals. Advances in Chemical Technique.	52/I
Power, B. D., *et al.*	Improvements i.o.r.t. Vapour Vacuum Pumps.	5/II
Preuss, L. E.	A Study of the Propagation Mode for Metallic Vapours in Shadow-Casting by Vacuum Evaporation of Au^{198} and Cr^{51}.	33/I
Puri, B. R., *et al.*	Boiling Point of Capillary-Condensed Water.	38/I
Ranken, M. B.	Moisture in Refrigerant Machinery Circuits and Components.	24/III
Ratcliffe, J. D.	Spare Parts for Humans.	16/I
Reasbeck, P., *et al.*	Cosmic-Ray Production of Helium in Meteorites and Their Ages.	53/III
Rhoads, H. U., *et al.*	Structure of Evaporated Films as a Function of Film Thickness.	11/III
Rob, C. G.	The Preservation of Arterial Grafts by Freeze Drying.	40/I
Roberts, C. C., *et al.*	Techniques for Measuring and Controlling Vacuum.	3/I
Robinson, P. H., *et al.*	Rapid Interferometric Analysis of Germane-Hydrogen Mixtures.	3/IV
Roehrig, J. R., *et al*	Wide-Range Vacuum Gauge.	8/II
Sawyer, P. N., *et al.*	Electrical Potential Differences Across the Normal Aorta and Aortic Grafts of Dogs.	41/I
Sayce, L. A.	The Production of Diffraction Gratings.	27/I
Scheer, K. E.	A Simple Laboratory for Freeze Drying.	7/IV
Scheuble, W.	Sorption Properties of Thin Nickel Films.	45/I
Schwager, J. E., *et al.*	Note on the Preparation of Thin Vanadium Targets.	54/III
Shepard, M.	Economical Coatings by Vacuum Metallising.	5/III
Sibley, C. B., *et al.*	Wide-Range Vacuum Gauge.	8/II
Sikorski, J., *et al.*	A Rotary Vacuum Seal.	29/II
Sims, R. F.	Wear of Carbon Brushes at High Altitudes.	21/I
Souza Santos, Th.D., de	Furnace for Vacuum Distillation of Zinc and Silver, Constructed by the Institute Pesquisas Tecnologicas.	32/III
Staunton, J. L.	Rust in Water Checked by Deaeration.	10/I
Stinnett, A. J.	Repairing Ionisation Gauge Tubes.	11/II
Stoddard, C. K., *et al.*	Production of Titanium Metal.	35/III
Stone, J. M.	Refinements in the Preparation and Theory of Multilayer Films.	28/I
Stone, N. W. B., *et al.*	The Influence of Barometric Pressure on Watch and Chronometer Rates.	15/I
Strickland, R. F., *et al.*	A Study of the Factors Governing the Rate of Freeze Drying of Blood Plasma.	42/I
Sutton, D. A.	A Three-Stage Molecular Still.	41/III
Taieb, J., *et al.*	A Few Remarks on the Electronic Circuit of a Leak Detector.	23/I
Thébault, J., *et al.*	A Few Remarks on the Electronic Circuit of a Leak Detector.	23/I
Thomson, S. J., *et al.*	Cosmic-Ray Production of Helium in Meteorites and Their Ages.	53/III
Trapnell, B. M. W.	The Activities of Evaporated Metal Films in Gas Chemisorption.	44/I
Treffenberg, L.	A Method of Freezing Drying of Histological Preparations.	8/IV
Trevoy, D. J.	Rate of Evaporation of Glycerol in High Vacuum.	39/I
Trolan, J. K., *et al.*	The Field-Emission Initiated Vacuum Arc. I. Experiments on Arc Initiation.	13/IV
Trolan, J. K., *et al.*	The Field-Emission Initiated Vacuum Arc. II. The Resistively Heated Emitter.	14/IV

Vol. 4 1954

Cumulative Author Index, Vol. IV (1954)
Contd.

Author	Title of Publication	Abstr. No.
U.S. Navy, *et al.*	Method of Testing a Metallic Sample.	60/I
Veldhuis, M. K., *et al.*	The Recovery of Essence from Florida Orange Juices.	46/III
Vogt, A.	Method and Equipment for the Deposition of a Metal Layer on Wire or Similarly Shaped Material.	14/III
Vogt, A.	Improvements i.o.r.t. Cleaning Observation Windows in High Vacuum Chambers.	31/III
Warren, D. T., *et al.*	Rotary Vacuum Reaction Chambers.	4/IV
Watt, W., *et al.*	Density of Deposited Carbon.	24/II
Webb, D. P. D., *et al.*	The Diffraction of 150 kV Electrons.	16/II
Weber, A. H., *et al.*	Structure of Evaporated Films as a Function of Film Thickness.	11/III
Wexler, A., *et al.*	Metal-to-Glass Seals for Sub-λPoint Helium Apparatus.	17/IV
Whitney, W. B., *et al.*	Apparatus for Vacuum Distillation.	42/III
Wilman, H., *et al.*	The Origin of Stress in Metal Layers Condensed from Vapour in High Vacuum.	7/III
Wilson, L. V., *et al.*	A Metal Vacuum Lock for a Mass Spectrometer.	13/II
Woods, H. J., *et al.*	A Rotary Vacuum Seal.	29/II
Wyatt, J. L., *et al.*	Production of Titanium Metal.	35/III
Yeater, M. L., *et al.*	Method of Testing a Metallic Sample.	60/I
Yamaguchi, S.	A ' Double ' Camera for Electron Diffraction.	18/II
Zimmermann, L.	Procedure for the Graphical Calculations of Multi-Stage Ejector Pumps.	3/II
Ziock, K.	Experience Gained with Various Leak Detection Methods and Apparatus.	5/I

Vol.
4
195

Abbreviations and Publishers of Journals Quoted in this Volume

A.B.M.	Associaco Brasiliera de Metais, Via Duto, Donna Paulina 80-8°, Andar (Palocio Maua), Sao Paulo, Brazil.
Acta Phys. Austriaca	Acta Physica Austriaca, Springer Verlag, Mölkebastei 5, Vienna, 1, Austria.
A.E.C. Rep.	U.S. Atomic Energy Commission Reports, Office of Technical Services, Department of Commerce, Washington 25, D.C., U.S.A.
Amer. J. Phys.	American Journal of Physics, American Institute of Physics, Michigan State College, East Lansing, Michigan, U.S.A.
Analyst	The Analyst, The Society of Public Analysts, 7-8, Idol Lane, London, E.C.3, England.
Analyt. Chem.	Analytical Chemistry, American Chemical Society, 20th and Northampton Streets, Easton, Pa., U.S.A.
Appl. Sci. Res.	Applied Scientific Research, Martinus Nijhoff, The Hague, Holland.
Ark. Zoologi	Arkiv for Zoologi, Almquist & Wiksells Boktryckeri AB, Stockholm, Sweden.
Brit. J. Appl. Phys.	British Journal of Applied Physics, Institute of Physics, 47, Belgrave Square, London, S.W.1, England.
Chem. Engng.	Chemical Engineering, McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 36, N.Y., U.S.A.
Chem. & Industr.	Chemistry and Industry, Society of Chemical Industry, 56, Victoria Street, London, S.W.1, England.
Chem. Ing. Tech.	Chemie Ingenieur Technik, Verlag Chemie G.m.b.H., Weinheim/Bergstrasse, Haupstrasse 129, Germany.
Chem. Technik.	Chemische Technik, VEB Verlag Technik, Unter Den Linden 12, Berlin, N.W.7, Germany.
Electronics	Electronics, McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 36, N.Y., U.S.A.
Endeavour	Imperial Chemical Industries Ltd., 26, Dover Street, London, W.1, England.
Engineer	The Engineer, Morgan Brothers (Publishers) Ltd., 28, Essex Street, Strand, London, W.C.2, England.
Eng. News Rec.	Engineering News Record, McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 36, N.Y., U.S.A.
Food Engng.	Food Engineering, McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 36, N.Y., U.S.A.
Food Technol.	Food Technology, Institute of Food Technologists, 119-123, West Park Avenue, Champaign, Ill., U.S.A.
Glas-und Hochvakuum Tech.	Glas und Hochvakuum Technik, Rudolf A. Lang Verlag, Leibnizstrasse 64, Berlin-Charlottenburg 4, Germany.
Industr. Engng. Chem.	Industrial and Engineering Chemistry, American Chemical Society, 20th and Northampton Streets, Easton, Pa., U.S.A.
Inst. Chem. Engrs.	Institute of Chemical Engineers, 56, Victoria Street, London, S.W.1, England.
Inst. Refrig.	The Institute of Refrigeration, Dalmeny House, Monument Street, London, E.C.3, England.
J. Appl Phys.	Journal of Applied Physics, American Institute of Physics, 57, East 55th Street, New York 22, N.Y., U.S.A.
J. Inst. Petrol	Journal of the Institute of Petroleum, 26, Portland Place, London, W.1, England.
J. Metals	Journal of Metals, American Institute of Mining and Metallurgical Engineers Inc., 29, West 39th Street, New York 18, N.Y., U.S.A.
J. Opt. Soc. Amer.	Journal of the Optical Society of America, American Institute of Physics, Prince and Lemon Streets, Lancaster, Pa., U.S.A.
J. Res. Nat. Bur. Stand.	Journal of Research of the National Bureau of Standards, U.S. Government Printing Office, Washington 25, D.C., U.S.A.
J. Sci. Instrum.	Journal of Scientific Instruments, Institute of Physics, 47, Belgrave Square, London, S.W.1, England.
Mater. & Meth.	Material and Methods, 330, West 42nd Street, New York 18, N.Y., U.S.A.
Metal Finish.	Metal Finishing, Finishing Publications Inc., 11, West 42nd Street, New York 18, N.Y., U.S.A.
Metal Progr.	Metal Progress, The American Society for Metals, 7301, Euclid Avenue, Cleveland 3, Ohio, U.S.A.

ol.
4
954

Abbreviations and Publishers of Journals Quoted in this Volume

Contd.

Metallurgia	Metallurgia, The Kennedy Press Ltd., 31, King Street West, Manchester 3, England.
Mod. Refrig.	Modern Refrigeration, Refrigeration House, Victoria Road, Woking, Surrey, England.
Nature	Nature, Macmillan and Co., St. Martin's Street, London, W.C.2, England.
Naturwiss.	Naturwissenschaften, Springer Verlag, Jebenstrasse 1, Berlin-Charlottenburg, Germany.
Phys. Rev.	Physical Review, American Institute of Physics, Brookhaven National Laboratory, Upton, Long Island, N.Y., U.S.A.
Proc. Inst. Electr. Engrs.	Proceedings of the Institution of Electrical Engineers, Institution of Electrical Engineers, Savoy Place, Strand, London, W.C.2, England.
Proc. Phys. Soc.	Proceedings of the Physical Society, The Physical Society, 1, Lowther Gardens, Prince Consort Road, London, S.W.7, England.
Proc. Roy. Soc.	Proceedings of the Physical Society, The Royal Society, Burlington House, Piccadilly, London, W.1, England.
Prod. Engng.	Product. Engineering, McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 36, N.Y., U.S.A.
Reader's Dig.	Reader's Digest Association Ltd., 27, Albemarle Street, London, W.1, England.
Rev. Mod. Phys.	Review of Modern Physics, American Institute of Physics, Lancaster, Pa., U.S.A.
Rev. Sci. Instrum.	Review of Scientific Instruments, American Institute of Physics, 57, East 55th Street, New York 22, N.Y., U.S.A.
Stahl u. Eisen	Stahl und Eisen, Düsseldorf, Germany.
Steel	Steel, Penton Publishing Co., Penton Building. Cleveland 13, Ohio, U.S.A.
Sylvan. Tech.	Sylvania Technologist, Sylvania Electric Products, Inc., Sylvania Center, Bayside, N.Y., U.S.A.
Times Sci. Rev.	Times Science Review, The Times, London, E.C.4, England.
Le Vide	Le Vide, Société Française des Ingénieurs des Techniciens du Vide, 44, Rue de Rennes, Paris 6e., France.
World Refrig.	World Refrigeration, 181, Kensington High Street, London, W.8, England.
Zentralblatt	Zentralblatt für Bakteriologie, Parasitenkunde, Infektionskrankheiten und Hygiene, VEB Gustav Fischer Verlag, Jena, Germany.
Z. Phys.	Zeitschrift für Physik, Springer Verlag, Jebenstrasse 1, Berlin-Charlottenburg, Germany.

Vol.
4
195